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# Liner Materials Exposed to Hazardous and Toxic Sludges

Matrecon, Inc, Oakland, Calif

Prepared for

Municipal Environmental Research Lab, Cincinnati, Ohio

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# LINER MATERIALS EXPOSED TO HAZARDOUS AND TOXIC SLUDGES

First Interim Report

by

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Contract 68-03-2173

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## FOREWORD

The Environmental Protection Agency was created because of increasing public and government concern about the dangers of pollution to the health and welfare of the American poeple. Noxious air, foul water, and spoiled land are tragic testimony to the deterioration of our natural environment. The complexity of that environment and the interplay between its components require a concentrated and integrated attack on the problem.

Research and development is that necessary first step in problem solution and it involves defining the problem, measuring its impact, and searching for solutions. The Municipal Environmental Research Laboratory develops new and improved technology and systems for the prevention, treatment, and management of wastewater and solid and hazardous waste pollutant discharges from municipal and community sources, for the preservation and treatment of public drinking water supplies, and to minimize the adverse economic, social, health, and aesthetic effects of pollution. This publication is one of the products of that research; a most vital communiciations link between the researcher and the user community.

Although the information contained herein is preliminary, it will provide a guide and insight to the effects that happen after limited exposure. This information and data could be useful for design purposes if not taken out of context.

Francis T. Mayo, Director
Municipal Environmental Rsearch
Laboratory

## ABSTRACT

The storage and disposal of hazardous liquids and solid wastes on the land are increasing the potential for pollution of surface and ground waters by these wastes or their leachates. Intercepting and controlling the seepage of such fluids by the use of impervious barriers offers a promising means of reducing or eliminating such pollution.

This engineering research project was undertaken to assess the relative effectiveness and durability of a wide variety of liner materials when exposed to hazardous wastes. The materials under study include a native soil, modified bentonite, a soil cement, a hydraulic asphalt concrete, an asphaltic membrane, and 8 polymeric membranes based upon polyvinyl chloride, chlorinated polyethylene, chlorosulfonated polyethylene, ethylene propylene rubber, neoprene, butyl rubber, an elasticized polyolefin, and a thermoplastic polyester elastomer, respectively.

In this study the liner materials are exposed to such hazardous wastes as a strong acid, a strong base, an oil refinery tank bottom waste, a blend of lead wastes from gasoline production, a saturated and unsaturated hydrocarbon oil waste, and a pesticide.

The experimental approach and methodology followed are described and results of preliminary tests used in the selection of materials for extensive testing are presented.

This report was submitted in partial fulfillment of Contract 68-03-2173 by Matrecon, Inc., under the sponsorship of the U. S. Environmental Protection Agency. This report covers a period from February 3, 1975, to October 31, 1976.

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## LIST OF ABBREVIATIONS

ABBREVIATIONS

| | |
|---|---|
| COD | -- chemical oxygen demand |
| CPE | -- chlorinated polyethylene |
| EPDM | -- ethylene propylene rubber |
| HAC | -- hydraulic asphalt concrete |
| ipm | -- inches per minute |
| NT | -- no test |
| OT | -- open tub |
| PE | -- polyethylene |
| ppi | -- pounds per inch |
| ppm | -- parts per million |
| psi | -- pounds per square inch |
| PVC | -- polyvinyl chloride |
| SERL | -- Sanitary Engineering Research Laboratory, University of California, Berkeley, CA. |
| THF | -- tetrahydrofuran |
| TVA | -- total volatile acids |

NOMENCLATURE FOR LOCUS OF FAILURE IN ADHESIVE TESTING

| | |
|---|---|
| AD | -- failure within the adhesive |
| AD-AD | -- failure between two coats of adhesive |
| AD-LS | -- failure between adhesive and liner surface |
| BRK | -- break of liner material outside of the seam |
| DEL | -- delamination of the liner material |
| LS | -- failure at liner surface |
| OR | -- failure of the reinforcing fabric |

FACTORS FOR CONVERTING DATA IN U.S. CUSTOMARY UNITS TO SI METRIC UNITS

| | |
|---|---|
| Inches to centimeters (cm) | x 2.54 |
| Feet to metres | x 0.3048 |
| Mils to centimetres (cm) | $x\ 2.54 \times 10^{-3}$ |
| Mils to millimetres (mm) | $x\ 2.54 \times 10^{-2}$ |
| Pounds per square inch (psi) to megapascals (MPa) | $x\ 6.895 \times 10^{-3}$ |
| Pounds per inch (ppi) to kilo Newtons per metre (kN/m) | $x\ 1.751 \times 10^{-1}$ |
| Pound (force) to Newtons | x 4.448 |

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## SECTION 1

## INTRODUCTION AND OBJECTIVES

Environmental Protection Agency's "Report to Congress on Hazardous Waste Disposal" in 1973 (1) concluded that the then existing current management of the nation's hazardous residues was "generally inadequate" and that the "public health and welfare are being unnecessarily threatened by the uncontrolled discharge of such waste materials into the environment". At that time it was pointed out that approximately 10 million tons of nonradioactive hazardous wastes were being generated per year, of which 60% were organic and 40% inorganic. Of these wastes 90% occurred in liquid or semiliquid form. Generation of hazardous wastes is growing at the rate of 5 to 10% per year. However, at the same time, disposal on land is increasing even faster as a result of air and water pollution controls which require that those wastes be converted into solid or semisolid form which must then be disposed of on land. Ocean-dumping is becoming unacceptable for many wastes.

This increasing storage and disposal of hazardous wastes on the land increases the potential for pollution and contamination of the surface and ground water system. Positive measures are needed to prevent such occurrences.

Confining and controlling of hazardous wastes through the use of manmade impervious liner materials appears to be a feasible means of preventing the seepage of pollutants from the wastes entering and polluting the ground water. Such methods have been used to varying degrees for impounding wastes for the last 25 years. A great range of materials (2-7), differing in permeability to water and potential wastes, composition, form and shape, and costs, have been used or are potentially useful for confining a range of potential pollutants. Nevertheless, available information as to the relative performance and service lives of specific materials exposed to specific wastes is meager. In order to be able to supply guidance and perhaps eventual regulation for the use of liners in these applications, the Environmental Protection Agency needs considerably more information.

This project was undertaken with the following broad objectives:

1. To determine the effects upon a selected group of liner materials of exposing them to various hazardous wastes over an extended period of time.

2. To determine the durability of and the cost effectiveness of utilizing synthetic membranes, admixed materials, and natural soils as liners for hazardous wastes storage and disposal ponds.

3. To estimate the effective lives of 12 liner materials exposed to 6 types of nonradioactive hazardous waste streams generated by industry under conditions which simulate those encountered in holding ponds, lagoons, and landfills.

4. To develop information needed for selecting specific liner materials for confining hazardous wastes in specific installations.

5. To develop a method for assessing the relative merit of the various liner materials for specific applications and for determining their service lives.

6. To aid the Environmental Protection Agency in developing effective controls for the proper disposal and management of hazardous wastes.

This first interim report describes:

1. The methodology and research approach.

2. The construction of the exposure cells and the ancillary equipment.

3. The selection of the various liners and wastes.

4. The preparation of the admix liners.

5. The properties of the unexposed liners.

6. A preliminary experiment in which the compatibility of liners and the wastes was determined as a part of the process to determine the combination of liners and wastes which should be tested.

## SECTION 2

## SUMMARY OF WORK AND TENTATIVE CONCLUSIONS

One hundred forty-four cells, in which the various liner materials can be exposed under conditions which partially simulate actual service conditions, have been fabricated. These cells were designed to accept both membrane and thick admix liner materials. The area of an individual specimen exposed to the waste is 10 by 15 inches; waste 1 foot deep can be placed on it. These cells are placed in a covered rack with each cell having an individual cover. In addition, 12 plywood troughs with sloping sides were fabricated for exposing liners under conditions which simulate those encountered around the edges of waste ponds and lagoons.

Five types of admix materials were selected for the exposure testing. These, with their respective thicknesses, are:

Asphalt emulsion on nonwoven fabric (0.3 in.).
Compacted native fine-grain soil (12.0 in.).
Hydraulic asphalt concrete (2.5 in.).
Modified bentonite and sand (5.0 in.).
Soil cement with and without surface seal (4.0 in.).

A total of 48 admix liner specimens have been placed in the cells.

Eight types of polymeric membrane liners were selected for exposure testing. These, with their respective thicknesses, are:

Butyl rubber, fabric reinforced (34 mils).
Chlorinated polyethylene (32 mils).
Chlorosulfonated polyethylene, fabric reinforced (34 mils).
Elasticized polyolefin (20 mils).
Ethylene propylene rubber (50 mils).
Neoprene, fabric reinforced (32 mils).
Polyester elastomer, experimental (8 mils).
Polyvinyl chloride (30 mils).

Eighty-four membrane liner specimens have been mounted in the exposure cells.

All membrane specimens were mounted with lap seams prepared either by the suppliers of the liners or by the contractor in accordance with procedures recommended by the suppliers. These seams are made by the same procedures

as used in the field except that, being prepared in the factory or in the laboratory, they are prepared under better conditions.

Six classes of hazardous wastes were selected for this study. They are:

Strong acid
Strong base
Waste of saturated and unsaturated oils
Lead waste from gasoline tanks
Oil refinery tank bottom waste (aromatic oil)
Pesticide waste

Twelve individual wastes were obtained and a preliminary characterization was made of each. Eleven of these were satisfactory; however, only 4 were available in sufficient quantity to fill the cells. The 3 individual lead wastes were combined to make a sufficient quantity of this class of waste. At the time the cells were filled, a sufficient quantity of the tank bottom waste to fill the primary exposure cells had not been obtained and considerable difficulty was encountered in obtaining such a waste; arrangements have now been made for delivery of 110 gallons of a waste of this type.

Preliminary exposure tests were run on the various liner materials in the various wastes in order to select combinations for long-term exposure. It was recognized that some of the liner materials would not be compatible with some of the wastes. In these preliminary tests, most of the membrane liners and all of the asphaltic materials either swelled or dissolved in the aromatic hydrocrabons. Several combinations were thus eliminated, such as the asphalts with oily wastes. It was also found that clays could only hold the acidic and caustic wastes for short periods of time. These combinations were dropped from the long term exposure tests.

At the present time 113 liner specimens are under exposure to 5 different wastes in individual exposure cells. Two additional exposure tests are also underway:

1. Small specimens (6 inches by 6 inches) of the membrane liners are being exposed to the weather on racks.

2. Larger specimens (4 feet by 4 feet) are being exposed to weather and selected wastes as liners of small open tubs filled with the wastes.

The results obtained to date on the limited bench-scale tests of liner materials in the various wastes demonstrate that:

1. Liners should be carefully selected for specific wastes.

2. Liner materials should be subjected to preliminary exposure tests in the wastes prior to selection of specific liners.

3. Oily wastes cannot be safely contained with asphalt-based liners.

4. Oily wastes, particularly those containing aromatic components, may pose special problems for polymeric membranes, except for those which are crystalline. The noncrystalline materials, such as the rubbers and PVC, swell to varying degrees in the oily wastes. Such swelling can be particularly damaging to seams based upon cements.

5. The bentonite liners, polymer modified bentonite, and perhaps many soils, may not be satisfactory for confining strong acids and bases and concentrated brines.

6. Wastes containing both aqueous and oily phases may also pose special problems because of the need of the liner to resist simultaneously two fluids which are inherently different in their compatibility with materials.

## SECTION 3

## PLANNED FUTURE WORK

1. Expose liner specimens to the wastes through April 1978 and perform the necessary monitoring of the primary exposure cells.

2. Continue the outdoor exposure tests of the liner specimens.

3. Complete the detailed analysis of the various wastes. This work is being done by the Sanitary Engineering Research Laboratory of the University of California, Berkeley.

4. Dismantle half of the primary cells after approximately one year and the remaining cells after two years of exposure, recover the liner specimens, and determine the effects that the exposure to the wastes has had on their physical properties and permeabilities.

5. Increase the number of liners and individual wastes in this investigation. Additional specimens of liners will be immersed in the 6 wastes now in the cells and in several wastes to be placed in additional cells. These liner specimens will be sufficiently large so that major physical properties which are being determined on the primary test specimens can be measured at 3 exposure times.

6. Prepare test specimens of various liners in the shape of bags in which an absorbent can be placed. Thus, specimens having only one side exposed to a waste can be immersed at different depths in a pond.

7. Tabulate and analyze liner specifications furnished by the manufacturers or suppliers to determine the various physical tests which are used by the industry. The relation of these specifications with field performance will be determined.

8. Determine the major components of polymeric liners which are being used in this test program. Analyses will include, at a minimum, ash, extractables, and specific gravity. Of particular importance are the filler, plasticizer, and polymer contents of all the major materials being studied under this contract. Additional analyses, e.g. elemental and infrared, will be performed as needed.

## SECTION 4

## OVERALL APPROACH AND RESEARCH PLAN

To meet the objectives of this project the overall experimental approach is to expose specimens of a variety of potential liner materials to selected hazardous wastes over a period of several years under conditions which simulate real service and to determine the changes in physical properties and permeability of these materials to the wastes with exposure time.

More specifically, the plan has been:

1. To select liner materials which are, or potentially could be, used for lining ponds containing hazardous wastes.

2. To design and construct exposure cells simulating the conditions under which liners would exist in a pond, which would act as permeameters to measure actual seepage, if any occurs.

3. To select a range of hazardous wastes of various types which would be encountered in industry.

4. To characterize the wastes fully so that the behavior of specific liners can be predicted for exposure to actual wastes in a given installation.

5. To perform exploratory tests of liners and wastes to determine their "compatibility" and suitability for long-term exposure tests.

6. To expose liner specimens of about one square foot in area which are sealed in the bottom of cells and covered with approximately one foot of waste. Field type seams would be incorporated in all polymeric membrane liners.

7. To measure the seepage of the wastes through the liners and to determine the composition of each fluid which is collected below the liner specimen.

8. To measure the properties of the liners as a function of exposure time of up to about 3 years.

9. To determine the changes in the properties of small specimens of a wide range of liner materials immersed in the same wastes.

10. To expose samples of selected liners to the weather and wastes simultaneously by placing in tubs partially filled with the wastes.

## SECTION 5

## DESIGN AND CONSTRUCTION OF EXPOSURE CELLS AND ANCILLARY EQUIPMENT

The exposure cells and the special equipment and materials required for this project are discussed in this section. They include the following items:

1. The primary exposure cells in which the liner membranes are being tested in contact with wastes.

2. The rack in which the loaded exposure cells are mounted.

3. The open tubs lined with selected membranes, filled with wastes, and exposed to the weather.

4. A highly inert silica gravel for filling the bases of the exposure cells.

### PRIMARY EXPOSURE CELLS

The individual exposure cells were designed to perform as permeameters and to be adaptable to liners of thicknesses ranging from 20 mils to 12 inches, as shown schematically by Figures 1 and 2, for membranes and thick liner specimens, respectively. They provide for a test specimen 10 by 15 inches in area and a volume of waste of approximately 1 cubic foot, at a depth of 1 foot. Photographs of the exposure cells for thick admix specimens and for membrane liners are shown in Figures 3 and 4, respectively.

144 cells were fabricated from 11-gauge steel, coated on the inside with a chemically resistant epoxy coating, Epoxy 3, and on the outside with a rust-preventative paint. (See Appendix A for details regarding materials used in constructing cells and mounting liner specimens.)

After coating the first 24 cells with paint brush and pad, it was decided that a more economical way to coat the remaining 120 cells was by spraying. A jig was constructed to facilitate the coating of the cell tank interiors. The cell bases were laid out next to each other on long tables so that 20 could be sprayed as a group.

The spraying of the primer presented no problem as its viscosity was within the medium range for paint. Spraying of the two-part epoxy coating, Epoxy 2, was somewhat more difficult; a quart of the mixed coating has a pot-life of 42 minutes at 77°F, and a viscosity of 30 poise, the consistency of a very heavy paint.

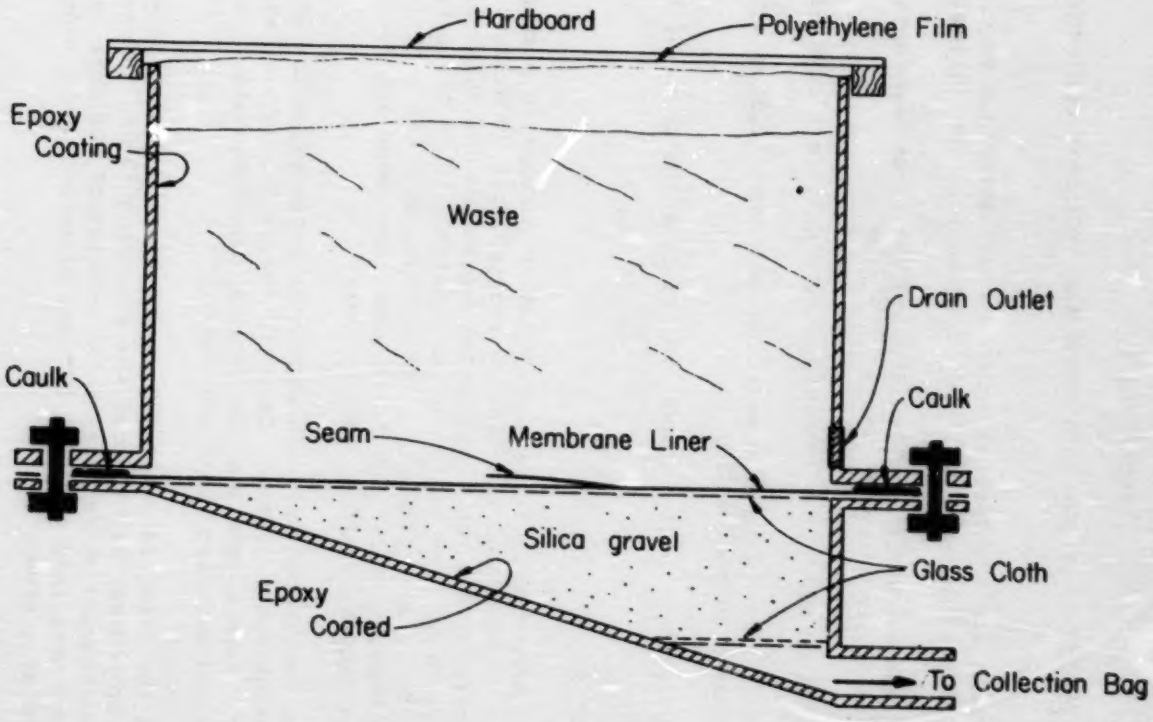



**Figure 1.** Schematic drawing of exposure cell for membrane liners. Exposed area of liner is 10 in. by 15 in. and depth of the waste is 1 foot.

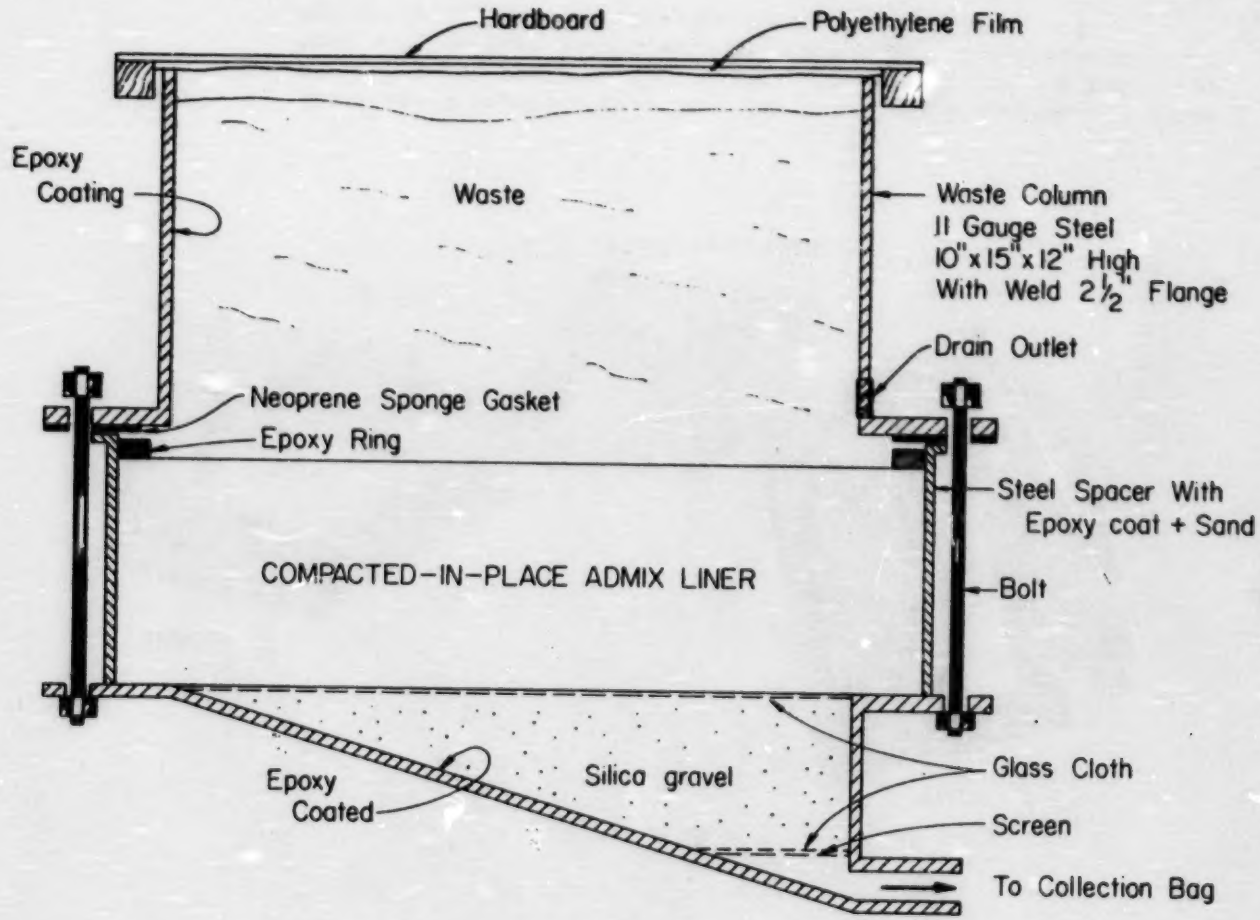



**Figure 2.** Schematic drawing of exposure cell for admix liners. Exposed area of liner is ca 12 in by 15 in and depth of the waste is 1 foot.

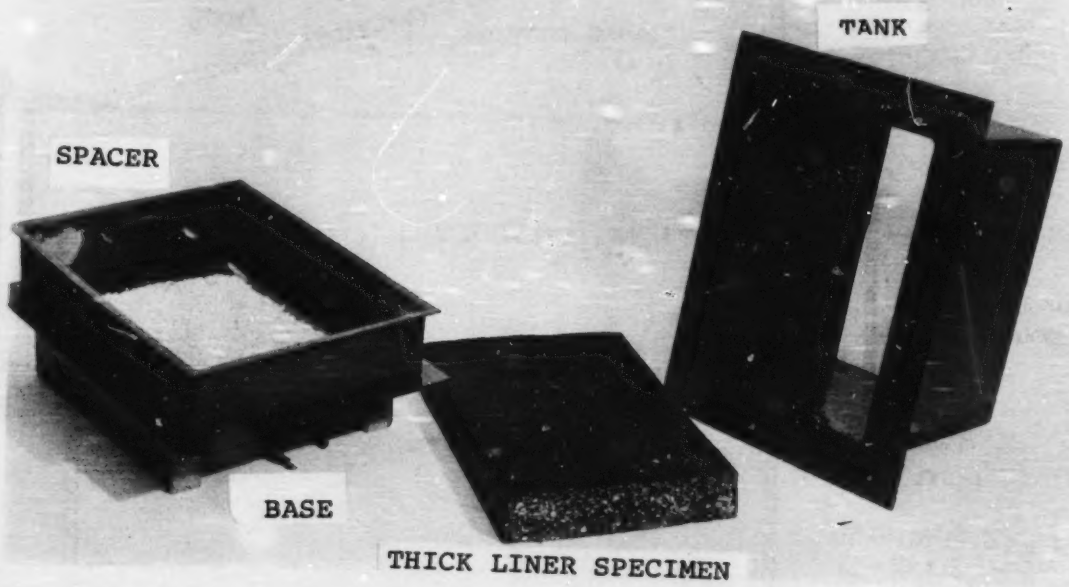



Figure 3. Unassembled exposure cell used for thick admix specimens. Shown are the three components, the tank, the spacer for the thick liners, and the base loaded with silica gravel. Shown also is an hydraulic asphalt concrete specimen.

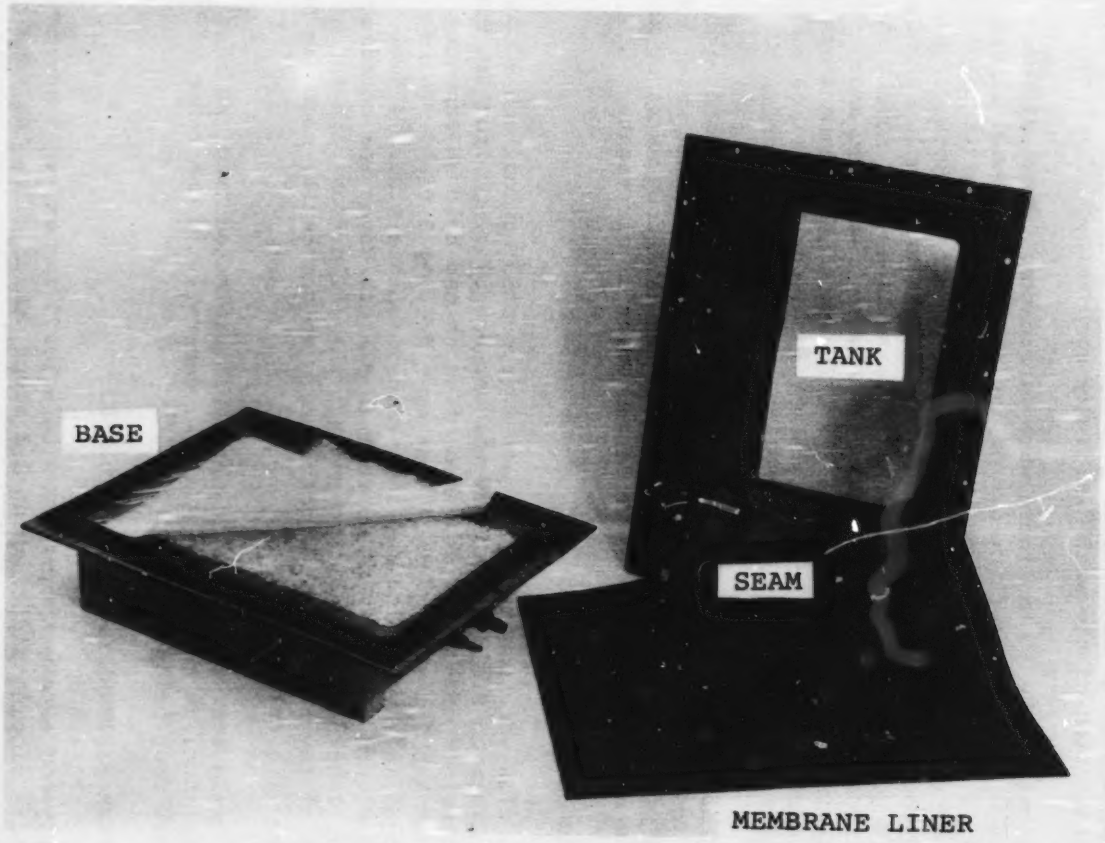



Figure 4. Unassembled exposure cell used for membrane liners. Shown are the tank and the base filled with silica gravel and a membrane liner specimen.

One coat of the primer was applied, followed by a coat of the two-part epoxy coating, Epoxy 2. A second coat of Epoxy 2 was applied along the bottom weld on the tank sections. All coatings were tested for pinholes with a spark tester. After 9 months of exposure there is no indication of failure or softening of the coating in any of the cells.

The bases of the cells were finished similarly to the tanks. They were filled with a chemically-inert high quality crushed silica in order to prevent reactions and contamination of the waste which may seep through the liner.

Spacers for mounting the admix liners were fabricated. These spacers varied in height depending on the thickness of the liner. For those liners compacted-in-place, i.e. the soil cement, native soil, and the modified bentonite, the inside surfaces were coated first with the primer and Epoxy 2, and then with a second coat of Epoxy 2 sprinkled with sandblasting sand. A rough wall surface should reduce or eliminate a wall effect which might result in the waste by-passing the liner. A ring seal of epoxy-sand was cast around the periphery of the admix specimens to increase path length of the waste seeping through the liner and to reduce wall effects and channeling. The rings were cast of Epoxy 1 and Epoxy 2 filled with sandblasting sand (See Appendix A).

Lids for each of the 144 cells were fabricated of hardboard, wood, and polyethylene, and installed on the filled cells. These lids reduce water loss by evaporation and prevent accidental contact with the hazardous waste by people or animals, but are not intended to be either water or air tight.

RACK FOR HOLDING EXPOSURE CELLS

The exposure cells were mounted on a rack constructed outdoors on a concrete slab at the Richmond Field Station of the University of California, Berkeley. This rack consists of 6 sections, each of which can hold 24 exposure cells. Each section has 2 tiers and is 4 feet by 8 feet by 3 feet. These 6 sections are arranged in 2 rows of 3 sections each which are placed 4 feet apart, as shown in Figures 5 and 6. A corrugated plastic cover was constructed over the rack at a height of 8 feet to protect the cells from rain and sunlight. The design of the racks affords good access to the cells; each can be removed or replaced with relative ease.

Although the cells are outdoors, they are well protected from the heat of the sun. The temperature is relatively uniform, generally in the range of 50 to 60 $^{o}$F, and does not drop below freezing.

OPEN TUBS FOR WEATHER EXPOSURE OF MEMBRANE LINERS

Twelve open plywood exposure tubs, 20 x 21 x 10.75 inches, were constructed. These tubs have sloping sides, about 1:2 slope, which allow the liners to be exposed to intermittent immersed-in-waste, exposed-to-weather cycles. Such conditions exist along the sides of operating hazardous waste ponds.

Since these plywood tubs are open to the weather, they require continual monitoring to prevent excessive evaporation, or overfilling from rain. They

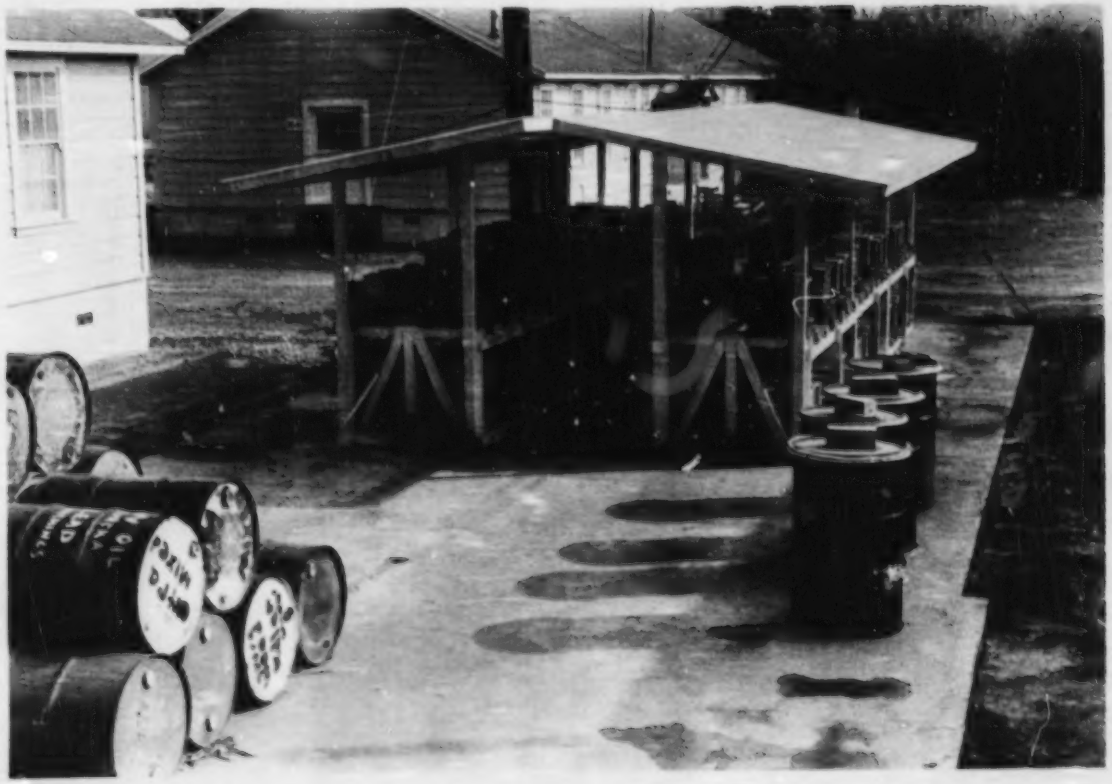


Figure 5. Overall view of the rack holding the exposure cells.

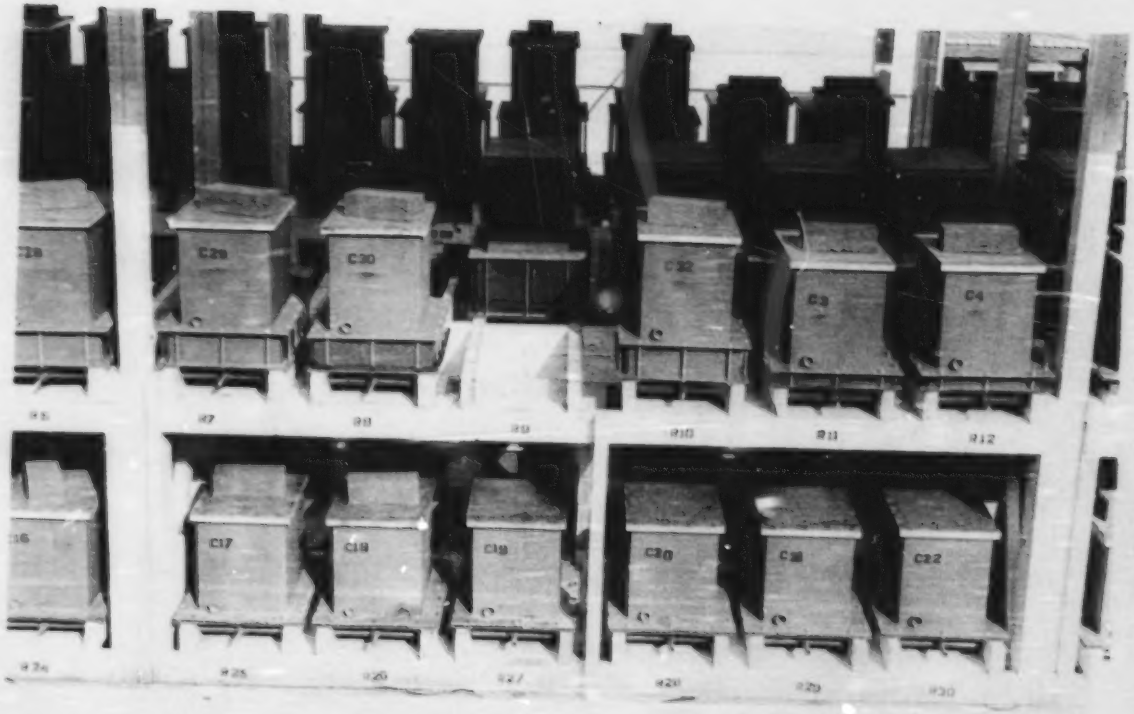



Figure 6. View of cells on the rack. These include both cells containing membrane and admix liners.

are placed in 2 shallow basins (8 feet by 6 feet by 4 inches) which can contain any accidental spills.

SILICA GRAVEL

The gravel used to fill the cell bases and support the liners was quartz from the Bear River (California), selected because of its high silica content (99%) and low content of constituents soluble in acid which might contaminate any of the wastes which percolate through the liners. The nominal size used was 3 to 6 mm (1/8 to 1/4 inch). Individual pieces were as long as 25 mm in one dimension, since the gravel had been crushed in a jaw crusher which tends to produce a high percentage of long and flat particles. The gravel was thoroughly washed and dried before placing in the bases.

## SECTION 6

## SELECTION AND PROPERTIES OF LINERS

A wide range of materials are potentially useful as barriers for impounding hazardous liquids and sludges (2-7). Many are currently being used to line ponds, reservoirs, lagoons, and canals to reduce or, if possible, eliminate the seepage of liquids into the ground. These liners vary considerably in permeability, durability, and cost. Selection of a liner for a given installation depends upon many factors, such as the character of the waste being confined, soil conditions, availability of materials, the level of performance required, the design life of the pond and cost.

Compacted soils, clays, soil cements, and asphaltic materials have found wide use for lining reservoirs, ponds, and other water-retention facilities. Many materials have limitations, particularly in respect to their permeability to other liquids, e.g. brines and oils.

The synthetic polymeric membranes are of particular interest because of their low permeability. However, they can vary considerably in physical and chemical properties, installation, overall performance, and costs. In addition, even for a given polymer, there can be considerably variation in the liners among producers due to compound, design, and manufacturing differences.

After reviewing the available liner materials, 5 admix type and 8 specific polymeric membrane materials were selected for exposure testing as liners in cells loaded with various wastes. These materials are listed in Tables 1 and 2.

TABLE 1. SOIL AND ADMIX LINER MATERIALS FOR HAZARDOUS WASTES

| Material | Thickness in inches |
|---|---|
| Asphalt emulsion on nonwoven fabric | 0.3 |
| Compacted native fine-grain soil | 12.0 |
| Hydraulic asphalt concrete | 2.5 |
| Modified bentonite and sand | 5.0 |
| Soil cement with seal | 4.0 |

TABLE 2. POLYMERIC MEMBRANE LINERS FOR HAZARDOUS WASTES

| Material | Thickness in mils |
|---|---|
| Butyl rubber, reinforced | 34 |
| Chlorinated polyethylene (CPE) | 32 |
| Chlorosulfonated polyethylene, reinforced | 34 |
| Elasticized polyolefin | 25 |
| Ethylene propylene rubber (EPDM) | 50 |
| Polychloroprene (neoprene), reinforced | 32 |
| Polyester elastomer | 8 |
| Polyvinyl chloride (PVC) | 30 |

ADMIX LINERS

The admix liners were selected or designed to yield permeability coefficients of $10^{-7}$ cm/sec or less. In the case of the soil cements and compacted fine-grain soils, a series of compositions was compacted in order to achieve the desired coefficient of permeability. Though water permeability was the primary test in evaluating admix materials, other tests appropriate to the specific materials were also used. The tests used in evaluating the admix materials are given in Table 3.

TABLE 3. TESTING OF ADMIX LINER MATERIALS

| |
|---|
| Water permeability - back-pressure permeameter (10). |
| Density and voids - ASTM D1184 and D2041. |
| Water swell - California Division of Highways 305. |
| Compressive strength - ASTM D1074. |
| Penetration of asphalt - ASTM D5. |
| Viscosity, sliding plate of asphalts - California Division of Highways 348. |

Sources of materials for admix liners are given in Appendix C. Details regarding the individual liner materials are presented below.

Hydraulic Asphalt Concrete

The mix design used for the hydraulic asphalt concrete (HAC) liner specimens called for dense-graded aggregate to ¼ inch maximum size and 9 parts of asphalt AR-4000 per 100 aggregate. The concrete was prepared in a hot-mix plant.

The one-half ton batch of hydraulic asphalt concrete was hauled 5 miles to the test site in an insulated trailer, raked into a form (10 feet by 3.5 feet by 2.25 inches), and levelled with a wooden screed. Temperature of the mix during placing was 345°F. Compaction was started when the mix had cooled to 275°F and was performed using a gasoline-powered "Jumping Jack" rammer with a 10 inch by 10 inch compacting foot. Compaction was continued for one-half hour, at which time the HAC began to stick to the compactor foot. The HAC was quite plastic, and some asphalt bled to the surface.

A one-gallon sample of uncompacted HAC and a one-quart sample of the AR-4000 asphalt (from the tank at the mixing plant) were retained for laboratory tests. After the compacted HAC had cooled overnight, 14 two-inch diameter cores were cut. Water permeability measured on 6 of the cores ranged from $2.8 \times 10^{-8}$ to $1.7 \times 10^{-9}$ cm/sec. (see Table 4). Later, the compacted pavement was cut with a diamond saw into specimens 11.75 inches by 17 inches to fit in the spacers for mounting in cells. The consistencies of the original asphalt and extracted asphalts, as measured by penetration values, are presented in Table 5. These data show the hardening of the asphalt which takes place during mixing and compaction.

TABLE 4. WATER PERMEABILITY OF HYDRAULIC ASPHALT CONCRETE FROM COMPACTED SLAB FOR PREPARING LINER SPECIMENS.

| Core | Location on Slab | Coefficient of Permeability cm/sec | in/yr |
|---|---|---|---|
| 5 | Near last end compacted | $6.6 \times 10^{-9}$ | 0.08 |
| 7 | Near last end compacted | $2.8 \times 10^{-8}$ | 0.34 |
| 12 | Near last end compacted | $2.1 \times 10^{-8}$ | 0.26 |
| 16 | Near first end compacted | $< 1 \times 10^{-8}$[a] | $< 0.12$[a] |
| 16 | Top slice removed | $1.7 \times 10^{-8}$ | 0.21 |
| 17 | Near first end compacted | $< 1.5 \times 10^{-8}$[a] | $< 0.19$[a] |
| 18 | Near first end compacted | ca $1.7 \times 10^{-9}$ | ca 0.02 |

[a]Because of long test runs the specimens were removed from test before constant values were obtained.

TABLE 5. HYDRAULIC ASPHALT CONCRETE[a] - PENETRATION OF ASPHALT[b] AT 77°F.

| | |
|---|---|
| Penetration of asphalt, unmixed, from tank to hot mix plant | 138 |
| Penetration of asphalt, extracted[c] from uncompacted HAC | 96 |
| Penetration of asphalt, extracted[c] from cores of compacted HAC | 80 |

[a]Dense-graded aggregate (¼ inch max), 100; asphalt 9.0.
[b]AR-4000, ASTM D5.
[c]Extractable 8.4 parts per 100 aggregate.

Each specimen was sealed into a 3.5 inch spacer. The specimen was first placed on a cell base which had been filled with silica gravel. A spacer was placed around the specimen and a wax-coated metal form was placed on top of it to form a ring. The space between the spacer, the liner, and the base was filled with a grout consisting of Epoxy 1 (2 Part A:1 Part B:3 sand). Grout of similar composition was used to cast the ring above the specimen.

The HAC liner specimens were prepared and placed in the cells early in the project. When they were tested with water prior to adding wastes (Figure 7), 5 were found to leak between the spacer and the epoxy seal, probably the result of difference in thermal coefficients of the steel and the asphalt concrete. Five of the asphalt concrete specimen-spacer combinations were chilled and the steel spacers were removed. The surfaces of the epoxy seal were roughened, and the new spacers were cast of Epoxy 2 around and on top of the epoxy seal. This was accomplished through the use of a wax-coated steel form and a wooden outer form. The cast epoxy spacers consisted of Epoxy 2 filled with sand (2 Part A:1 Part B:4.5 sandblasting sand) with a thin layer of unfilled Epoxy 3 poured on top to form a level surface. Neoprene sponge gaskets were used to seal the joints between the spacers and exposure cell tanks as in all the other cells.

Emulsified Asphalt on Nonwoven Fabric

Specimens of emulsified asphalt applied on a nonwoven polypropylene fabric mat were furnished by the supplier in 18 inch by 24 inch sheets, 0.3 inch thick, with a seam running across the width at the center. These sheets were cut to a size which would fit loosely inside the 1.25 inch spacer.

When mounting this liner, the cut sheet was placed on a base filled with crushed silica gravel; the spacer was placed around the liner, and an epoxy ring was cast around the inside of the spacer. The epoxy resin was spread on, around, and slightly under the edge of the liner. This ring of epoxy seals the liner to the base and the spacer, so that no waste can by-pass the liner. The epoxy ring was a grout consisting of sand-filled epoxy resin (Epoxy 1:2 Part A:1 Part B:3 sand, and 2% thixotropic agent). The assembled cells were leak tested by filling with water; none of the seals leaked. (No leak has developed in any of these cells during the first 9 months of exposure.)

Compacted Natural Fine-grain Soil

Selection of soil--

An extensive search was required for native fine-grain soils which would have the low level of permeability desired and minimal interaction with the wastes. Several fine-grain soils, at various water contents, were evaluated in the laboratory by measuring the permeability of compacted specimens with the back pressure permeameter (10). It was desired to determine the minimum permeability which could be obtained with a given soil. The results are shown in Table 6.

The soils included materials used for "mudjacking" of portland cement concrete pavement, clays used for constructing tennis courts and baseball diamonds, a soil used to line a waste pond at a power plant in Nevada, and a

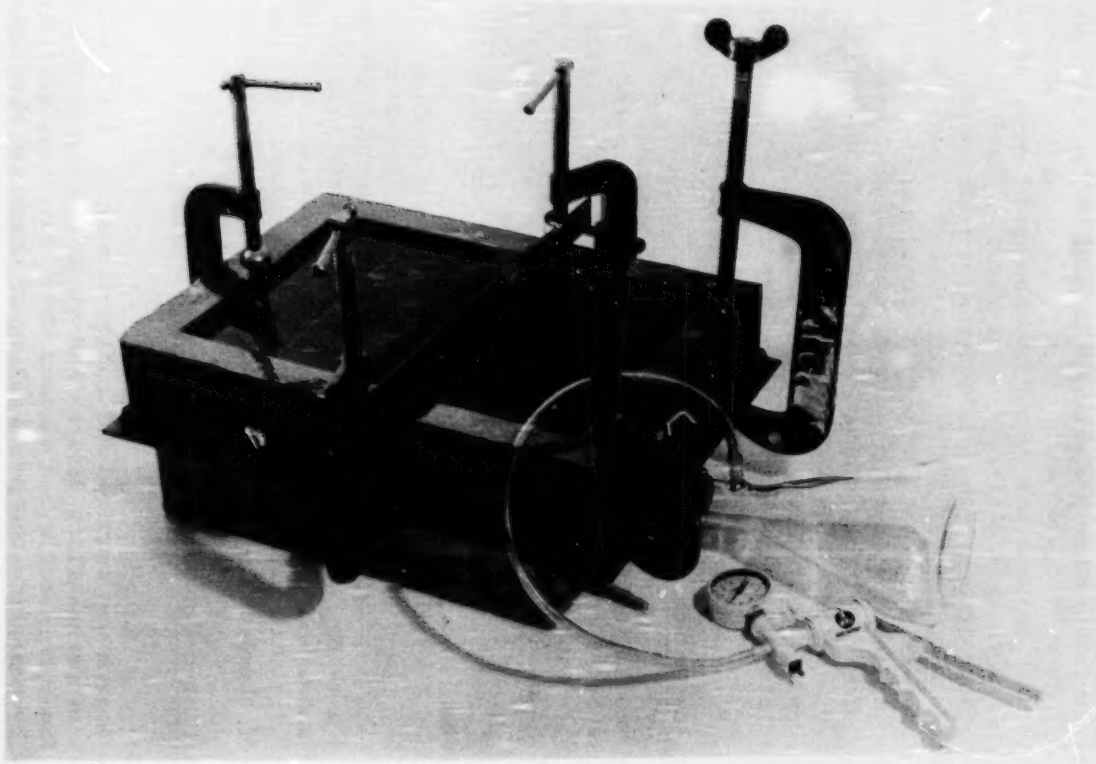


Figure 7. Leak-testing of hydraulic asphalt concrete. Specimen is covered with water and the base pressurized.

"Waste fines" which had been used to construct an impermeable cover over a chemical waste dump. (The last soil was selected for the soil cement.) The soil selected for use in the hazardous waste cells is a very fine silt that had been dredged from the Carquinez Straits and the lower Napa River and obtained at the Mare Island Naval Shipyard, Vallejo, California. Permeability of cores cut from the water-laid deposits was between $10^{-7}$ and $10^{-8}$ cm/sec. Permeability in the same range was measured on laboratory-compacted specimens prepared at water contents ranging from 14 to 30 parts of water per 100 parts dry soil (Table 6). Sieve analysis of the Mare Island soil showed more than 98% passing the 200 mesh sieve (75 $\mu$m) and almost 95% passing the 325 mesh sieve (45 $\mu$m).

TABLE 6. WATER PERMEABILITY OF LABORATORY COMPACTED NATIVE SOILS

| Soil[a] | Water,[b] parts per 100 dry soil | Coefficient of Permeability cm/sec | Coefficient of Permeability in/yr |
|---|---|---|---|
| Topsoil King clay | 10 | $4.0 \times 10^{-5}$ | 497 |
| Topsoil King "mudjacking" | 10 | $1.8 \times 10^{-5}$ | 223 |
| Topsoil Ling "mudjacking" | 11 | $7.9 \times 10^{-6}$ | 98 |
| Moapa, Nevada, #2 | 15 | $1.7 \times 10^{-7}$ | 2.10 |
| Moapa, Nevada, #2 | 17 | $4.1 \times 10^{-8}$ | 0.51 |
| Quarry Products,"wastes fines" | 12 | $4.9 \times 10^{-6}$ | 61.0 |
| Quarry Products,"wastes fines" | 15 | $2.8 \times 10^{-7}$ | 3.5 |
| Mare Island | 30 | $8.2 \times 10^{-8}$ | 1.02 |
| Mare Island | 28 | $7.8 \times 10^{-8}$ | 0.97 |
| Mare Island | 25 | $9.0 \times 10^{-8}$ | 1.12 |
| Mare Island | 20 | $6.5 \times 10^{-8}$ | 0.81 |
| Mare Island | 18 | $7.3 \times 10^{-8}$ | 0.91 |
| Mare Island | 16 | $1.1 \times 10^{-7}$ | 1.37 |
| Mare Island | 14 | $1.2 \times 10^{-7}$ | 1.49 |

[a] Carbonate content of soils: Quarry Products soil contains some carbonate; Moapa soil contains more carbonate; Mare Island soil contains no carbonate.
[b] Soils mixed with various amounts of soil, compacted, and tested to determine water content to achieve minimum permeability.

Preparation of Compacted Soil Liners in Cells--

The 13 inches high spacers, coated with primer, Epoxy 2, and sandblasting sand, were used for holding the native soil liners.

An attempt was made initially to compact the soil in a spacer which had been bolted on the base, but the assembly was too flexible; the bottom of the base and the sides of the spacer flexed with each impact of a tamper. The

bond between the soil and the spacer was not tight. Also, it was felt that the lowest potential water permeability was not achieved. Therefore, a wooden frame was constructed (Figure 8) in which the spacer was held tightly to prevent bulging of the sides and the whole assembly was bolted to a concrete floor. A piece of polyetheylene film was placed on the floor and the soil compacted on it. This film was removed before the liner was mounted in the base.

The 3 tampers used in compacting this soil, as well as the soil cement and clay specimens, are described below:

Tamper 1: Weight, 6.8 kg (15 lb)
Face, flat square, 125 x 125 mm (5 x 5 in)

Tamper 2: Weight 7.3 kg (16 lb)
Face A, flat round, 50 mm diam. (2 in)
Face B, "sheep's foot", 13 mm diam (0.5 in)

Tamper 3: Face, flat rectangular, 20 x 40 mm (0.75 x 1.5 in)
Struck with 1 kg hammer (2 lb)

Because of its plastic nature the Mare Island soil could not be blended with the soil-shredder as was done with the soil used for the soil-cement liners. Near or above its optimum moisture content, the soil was too sticky to pass through the shredder and, when drier, it was too hard causing the shredder to stall. Therefore, soil for the liners was blended by taking small scoopfuls from each of the bags of soil. Lumps that were dry or unusually wet were discarded. Soil for each lift (1.5 to 2 inches loose, compacted to 1 to 1.25 inches) was placed in the spacer, crushed and levelled with Tamper 1 and compacted by repeated tamping with Tamper 2 using alternately a 2 inch diameter flat face "A" and a ½ inch diameter "sheep's foot" face B. Final compaction of each lift was with the "sheep's foot" to provide a rough surface for keying to the next layer. The edges were compacted with Tamper 3 struck with a hammer, twice around all edges for each lift. The top layer of each liner was levelled, after compacting, by additional tamping with Tamper 1. The compacted soil liner is shown in Figure 9. These liners were covered with polyethylene film and protected from drying until mounted in the cells.

The cell bases were filled with the silica gravel levelled to support the soil. After the liners were mounted in the cells a ring of Epoxy 1 was cast into a triangular groove cut into the soil around the periphery of the liner. The outside of the joint between the spacer and the cell was sealed with butyl caulk.

Permeability measured on cores cut from compacted soil liners was slightly more permeable than the cores from the water-laid deposits in place at Mare Island. The permeability meaurements on the compacted liners are shown in Table 7.

## Soil Cement

### Selection of Soil--

Most of the fine-grain soils being considered for use as compacted native soil liners were evaluated in soil cement mixes. Formulations containing

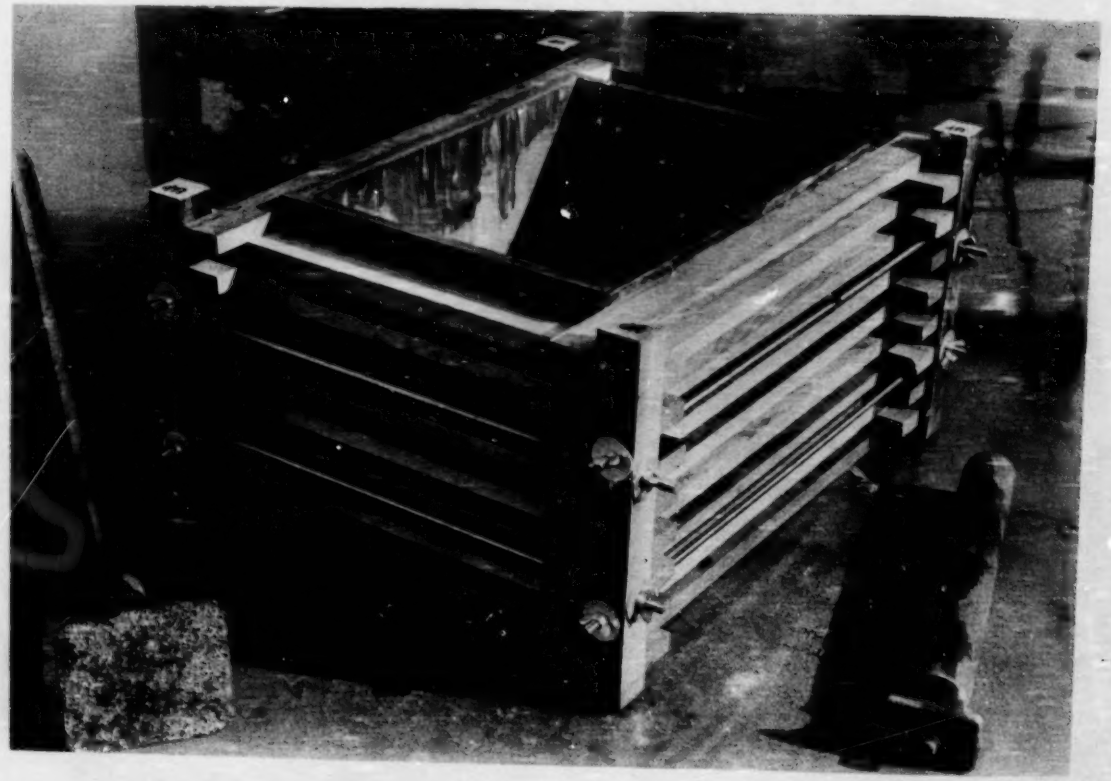


Figure 8. Jig used in compacting the specimens of soil and of other admix materials. It is bolted to the floor during compaction. The spacer for the soil specimens fits in this jig as assembled.

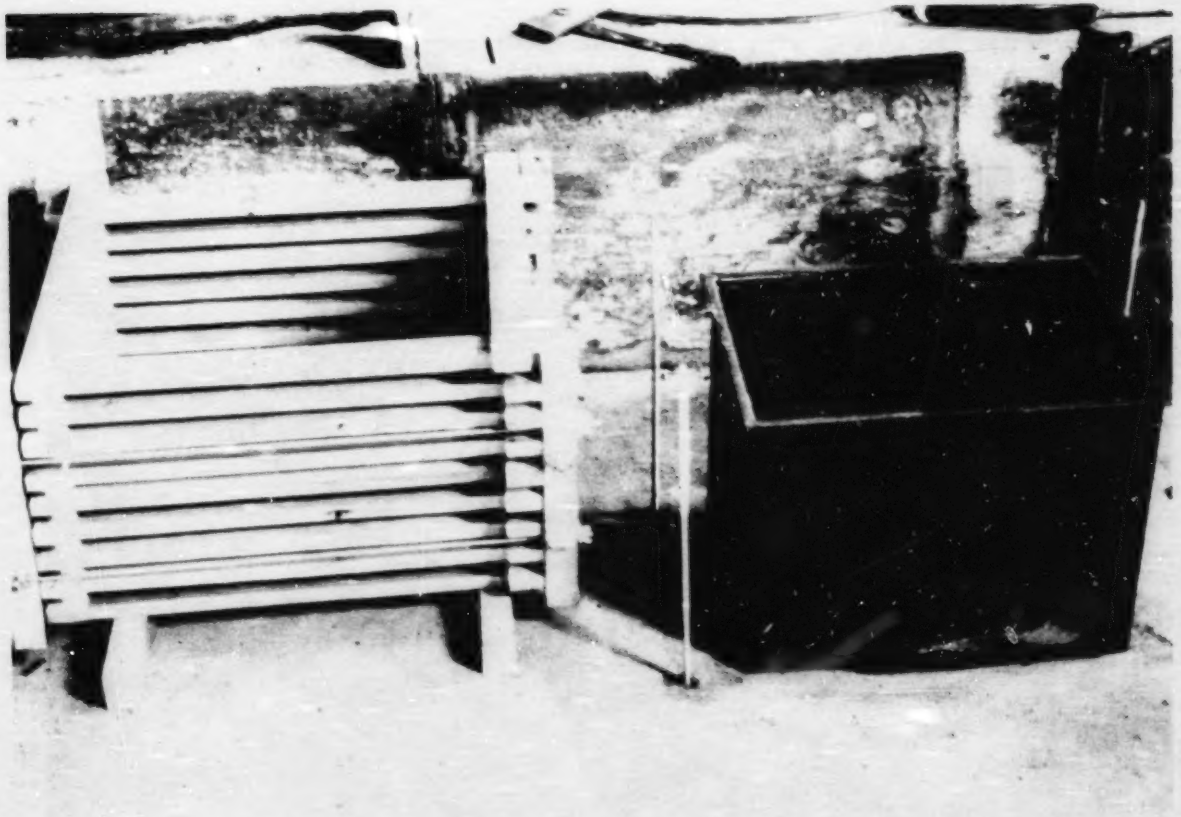


Figure 9. A compacted fine-grain soil liner specimen in 13 inches tall spacer ready to be mounted in cell base. Also shown are the frame in which the specimen had been compacted and bolts for holding the assembly to the floor while the liner was compacted.

TABLE 7. PERMEABILITY OF COMPACTED LINER SPECIMENS OF FINE-GRAIN SOIL FROM MARE ISLAND

| Source of Specimen | Coefficient of Permeability, cm/sec. |
|---|---|
| Laboratory-prepared specimens (7 samples)[a] | $6.5 \times 10^{-8}$ to $1.2 \times 10^{-7}$ |
| Core from specimen compacted in spacer on cell base | |
| Core from resilient end of cell | $4.5 \times 10^{-7}$ |
| Core from firm end of cell | $1.7 \times 10^{-7}$ |
| Core from 12-inches thick specimen compacted in spacer in jig on concrete floor (2 samples) | $9.4 \times 10^{-8}$ and $4.2 \times 10^{-7}$ |
| Core taken from water-laid deposits of the soil (3 samples) | $1.0 \times 10^{-8}$ to $1.0 \times 10^{-7}$ |

[a] See Table 6.

various proportions of soil:portland cement:water were molded into briquets, cured, and tested in the back-pressure permeameter. The lowest permeability was obtained with soil cement based on "waste-fines" from a local quarry, with 12 parts of Type 5 (sulfate-resistant) portland cement per 100 soil, and 12% of water on the total of soil plus cement (see Table 8). The "waste fines" material is available in large quantities as a by-product of the washing of crushed graywacke and has been used as a compacted soil cover (not a soil cement) over a chemical waste dump. Sieve analysis (wash) of the soil was:

| Sieve size | | % Passing |
|---|---|---|
| Mesh | Opening, μm | |
| 50 | 300 | 98 |
| 100 | 150 | 93 |
| 200 | 75 | 83 |
| 325 | 45 | 70 |

The "wastes fines" soil was received mostly in large friable lumps that were broken up by passing the soil twice through a shredder to yield a thorough blend with maximum agglomerate size of approximately 1/8 inch (3 mm). The soil was air-dried on a concrete slab then stored indoors until used.

Preparation of Compacted Soil Cement Liners--

The soil cement specimens were compacted in 5-inch high spacers; the inner surfaces were coated with epoxy and sand. The compaction procedure was the same as used with the fine-grain soil; the spacer was placed within a supporting wooden frame (Figure 8) to prevent bulging of the sides and the frame and spacer were bolted to a concrete floor. A piece of polyethylene film was placed on the concrete floor at the bottom of the spacer.

For each liner, 50 lb. of dry soil was thoroughly mixed in a mortar boat with 6 lb. of Type 1-2-5 portland cement, then with 6.7 lb. of water. The mixed soil-cement was crumbly and barely damp before compaction. One-inch

TABLE 8. WATER PERMEABILITY OF SOIL CEMENT SPECIMENS

| Composition | | | Coefficient of permeability | |
|---|---|---|---|---|
| Soil | Type V cement parts | Water parts | cm/sec. | in/yr |
| Topsoil King clay | 8 | 10 | $1.6 \times 10^{-6}$ | 20 |
| Topsoil King clay | 10 | 10 | $1.3 \times 10^{-6}$ | 16 |
| Topsoil King clay | 12 | 10 | $5.1 \times 10^{-6}$ | 63 |
| Topsoil King clay "mudjacking" | 8 | 12 | $3.4 \times 10^{-6}$ | 42 |
| Topsoil King clay "mudjacking" | 10 | 12 | $5.3 \times 10^{-6}$ | 66 |
| Topsoil King clay "mudjacking" | 12 | 12 | $6.5 \times 10^{-6}$ | 81 |
| Quarry Products soil | 10 | 13 | $1.9 \times 10^{-6}$ | 24 |
| Quarry Products soil | 12 | 12 | $1.5 \times 10^{-7}$ | 1.9 |
| Quarry Products soil | 10[a] | 12 | $2.9 \times 10^{-7}$<br>$4.0 \times 10^{-7}$[b] | 3.6<br>5.0 |
| Core from specimen compacted in spacer in cell base | 12 | 13.4 | $5.7 \times 10^{-8}$ | 0.71 |

[a] Rice hull ash cement (an acid-resistant cement).
[b] Repeat.

lifts were placed in the spacer and compacted with Tampers 1, 2, and 3, described previously. Figure 10 shows the compaction of the soil cement with Tamper 1. Final compaction of each lift was with the "sheep's foot" Face B of Tamper 2, to provide a rough surface for keying to the next layer. The edges were compacted with Tamper 3, twice around all edges for each lift. After a total compacted thickness of 4 inches was obtained, the top surface was levelled by additional tamping with Tamper 1. The compacted soil cement was quite firm, but plastic enough to be compacted without cracking or shattering. The thickness of the compacted liner specimens was 3.75 to 4.0 inches. One-half gallon of water was poured on the surface and the soil cement was cured for at least 7 days.

The surfaces of the cured liners were then allowed to dry only enough to provide good adhesion for an epoxy ring cast around the periphery of the liner, sealing the liner to the spacer, and for application of two coal-tar modified epoxy surface sealing coatings to portions of the liner surfaces. After the epoxies cured, the liners were again flooded with water until being filled with wastes. Each spacer was mounted on a cell base which had been filled with crushed silica gravel levelled to support the liner. Neoprene sponge gaskets were used to form seals between the flanges of the spacers and the cell tanks. After the cell base, the spacer with sealed-in liner, and the exposure tank were bolted together, a bead of butyl caulk was applied to the outside of the base-to-spacer junction to make it airtight.

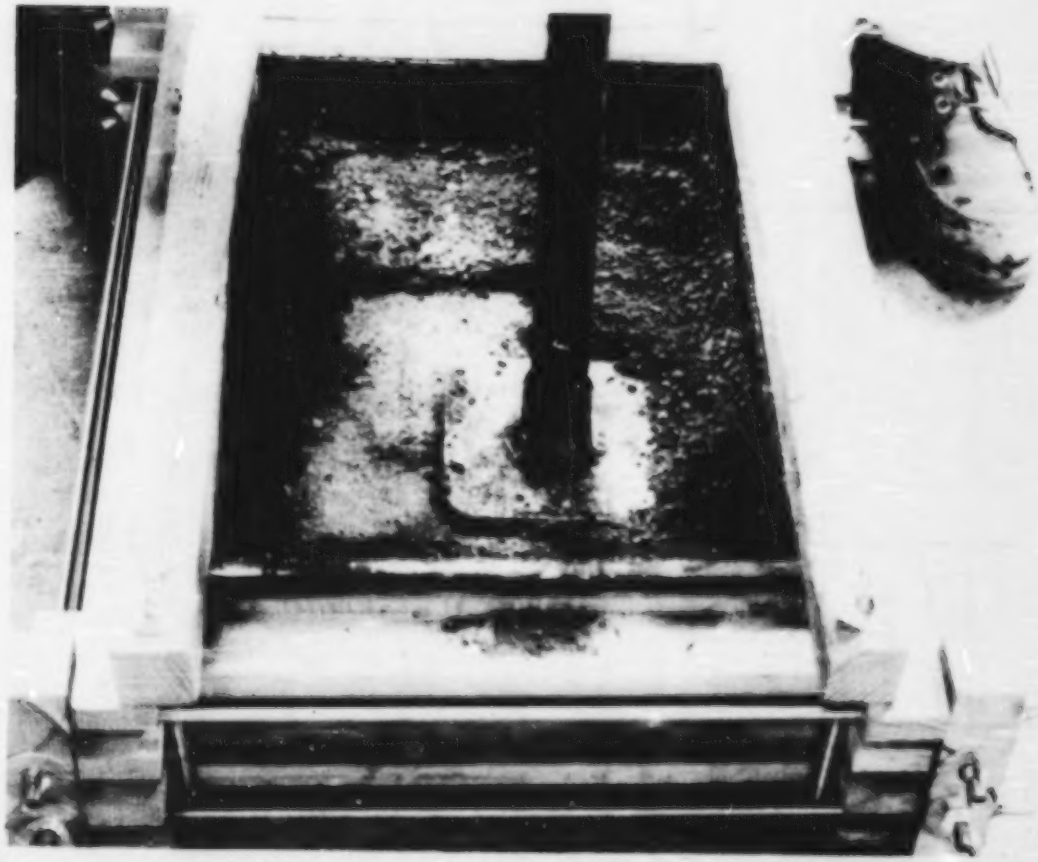
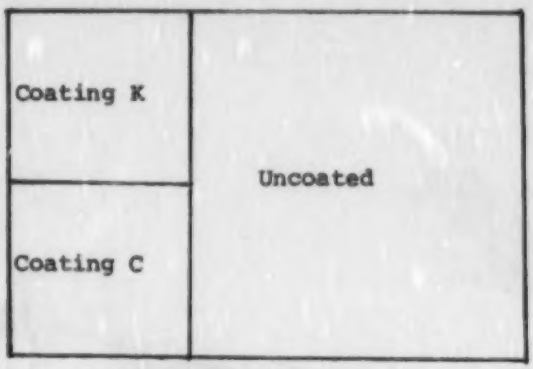


Figure 10. Compaction of a soil-cement liner specimen with Tamper 1 is shown as is the character of the uncompacted soil cement.

Application of Surface-sealing coatings--

Two sections of each soil-cement liner were coated with two coal-tar epoxy resin sealers. These sealers and the primer used for one of them were 2-component systems. Each was mixed according to the manufacturer's directions, and applied by brush. The areas coated were 6 inches square, located at one end of the cell, as shown below.




Coating K = 3 coats proprietary epoxy bituminous coating (first coat diluted, 2 parts:1 part thinner, according to manufacturer's instructions), average total thickness 0.044 inch.

Coating C = 1 coat primer, 0.007 inch, plus 2 coats proprietary epoxy-tar coating, 0.018 inch; total thickness 0.025 inch.

Treated Bentonite Clays

Selection--

Two polymer modified bentonite clays, claimed by their respective suppliers to be more effective than unmodified bentonite in the presence of dissolved salts, were included in the study. Laboratory specimens were prepared with 6%, 12%, and 20% of each treated bentonite in a well-graded sand, and briquets were compacted with various water contents to determine optimum moisture content for compaction to maximum density. Sieve analysis of the sand used was:

| Sieve size | | % Passing |
|---|---|---|
| Mesh | Opening, m | |
| 8 | 2360 | 96 |
| 16 | 1180 | 80 |
| 30 | 600 | 50 |
| 50 | 300 | 15 |
| 200 | 75 | 2.6 |

As the sand-clay compositions were not strong enough to be tested without support, specimens at the optimum moisture content were compacted in brass tubes for testing in the back-pressure permeameter. Permeability coefficients were in the range of $10^{-7}$ to $10^{-10}$ cm/sec, as shown in Table 9.

TABLE 9. WATER PERMEABILITY OF LABORATORY COMPACTED MODIFIED BENTONITE-SAND SPECIMENS.

| Composition | | | Coefficient of Permeability, cm/sec. | |
|---|---|---|---|---|
| Sand | Water | Bentonite | Clay A | Clay B |
| 94 | 12 | 6 | $5.0 \times 10^{-7}$ | $7.1 \times 10^{-8}$ |
| 88 | 12 | 12 | $7.4 \times 10^{-9}$ | $2.8 \times 10^{-9}$ |
| 80 | 12 | 20 | $8.0 \times 10^{-10}$ | $3.5 \times 10^{-9}$ |

Preparation of Treated Bentonite Liner Specimens--

The polymer modified bentonite liner specimens were compacted in the 7-inch high spacers. Each spacer was placed on a cell base previously coated with Epoxy 2; the outer flange of the base was slightly roughened with sandpaper to insure good adhesion of the epoxy. Masking tape was pressed around the flange between spacer and base, and a wax-coated metal form was placed on the base inside the spacer. Between the form and the spacer an epoxy seal, approximately ¼ inch high by 1 inch wide, was cast using the grout of the Epoxy 2 and sandblasting sand. After the seal was cured and the form removed, the base was filled with crushed silica gravel to the level of the bottom of the seal. The gravel was covered with woven glass fabric and the wooden reinforcing frame was bolted around the spacer. The clay-sand mixtures were compacted in the spacer following the same procedure and with the same tampers used in compacting the fine-grain soil.

Eight liner specimens were fabricated using 12% Clay B, 88% well-graded sand, mixed at optimum moisture content and compacted in the liners already sealed to bases. For each liner, 53.5 pounds of sand containing 3% water was mixed in a mortar boat with 5.5 pounds more water and with 7.1 pounds Clay B. This mixture was placed in the prepared spacer in 1-inch lifts, and each lift was compacted with tampers. The compacting effort was less than that used for the native soil and soil cement liners, to avoid damage to the epoxy seal between the spacer and the base.

Two liner specimens were fabricated using 20% Clay A and 80% well-graded sand mixed at 3% water content. They were also prepared in spacers.

The compacted Clay B liners were 5 inches thick and the dry-tamped Clay A liners 4 inches thick. Both were about 6 inches thick after complete hydration.

A sealing ring of Epoxy 2 grout was cast around the top edge of each of the compacted specimens.

Two gallons of tap water was added on each liner through a perforated can to avoid displacing the tamped sand-clay. After one day thickness increased from 4.0 to 4.5 inches and after one week to 4.75 inches. One week was allowed for "fresh-water hydration", as per manufacturers recommendation, before draining the excess water and adding wastes.

## POLYMERIC MEMBRANE LINERS

Flexible polymeric membranes are assuming increased importance as liner materials because of their very low permeability to water and other fluids. These liners are products of the plastics and rubber industries. The polymeric materials used in the manufacture of these liners include vulcanizable and nonvulcanizable (thermoplastic) plastics and rubbers. They are all synthetic materials, varying from highly polar polymers, such as polyvinyl chloride, to nonpolar polymers, such as EPDM and butyl. They range from amorphous polymers, such as the rubbers, to crystalline polymers, such as polyethylene. Generally, polymeric materials are compounded with fillers, antidegradants, plasticizers, and, if vulcanization is needed, curatives. Compounds based on the same polymer can vary considerably in composition from manufacturer to manufacturer, depending on the grade and the price of the liner.

The membrane sheeting is usually made in a continuous process by plying together two thin sheets formed by passing the compound through the rolls of a calender. Plying two sheets together to make a membrane almost eliminates pinholes. Fabric reinforcement, usually a nylon or polyester scrim, can be sandwiched between these plies to give added strength to the liner. Sheets are typically 4 to 5 feet wide and 200 feet long. Several of these sheets are seamed by a fabricator in a factory to form a panel.

These panels are brought to the site and seamed in the field to make the final liner. Field seaming is one of the major problem areas in the use of polymeric liners. Heat sealing, cementing, and solvent welding are used both in the factory and in field seaming. Seaming of vulcanized sheetings have presented the most problems, particularly in the field as cold-curing adhesives are required.

In making the selection of specific membrane materials for exposure tests, consideration was given to the following:

1. The need for a broad representation of polymer types in order to assure compatibility of liners and wastes, because of the great diversity in the composition of wastes.

2. Availability of several membrane liners which are under investigation for resistance to sanitary landfill leachate (4,8,9). They offer promise for impounding hazardous wastes and allow an intensive study on the characteristics of a few liners.

3. Equal thickness for liners, if possible. Most of the liners are available in 30 mil thicknesses; however, if this thickness is not available for the specific membrane then the thickness normally manufactured would be used.

In making the selection it was not possible to obtain liners from all liner producers. Representative liners of the respective types were selected. If several membranes of a given polymer were available, the membrane exhibiting the best physical properties was generally selected.

The specific polymer types which are included in the major portion of this study are discussed below.

Butyl Rubber

Butyl rubber is a copolymer of a major amount of isobutylene (97%) and a minor amount of isoprene to introduce unsaturation in the rubber as sites for vulcanization. A vulcanized butyl rubber compound is used in the manufacture of the sheeting, which is available in either unsupported or fabric-reinforced versions of 20 to 125 mil thickness. Butyl rubber has excellent resistance to permeation of water and swelling in water. This rubber has poor resistance to hydrocarbons, but is quite resistant to animal and vegetable oils and fats. Butyl rubber compounds generally contain low amounts of extractable material and swell little in water. Overall they age very well, although some butyl compounds ozone crack. Some recent compounds contain minor amounts of EPDM to improve ozone resistance. In outdoor exposure in water management use, butyl rubber liners have shown no degradation after 20 years of service. Obtaining good splices of butyl sheeting, particularly in the field, continues to be a problem, as cold curing adhesives are required.

Chlorinated Polyethylene (CPE)

This relatively recently developed polymer is an inherently flexible thermoplastic produced by chlorinating high density polyethylene. Sheeting of CPE makes durable linings for waste, water, or chemical storage pits, ponds, or reservoirs. CPE withstands ozone, weathering and ultraviolet and resists many corrosive chemicals, hydrocarbons, microbiological attack, and burning. Compounds of CPE are serviceable at low temperatures and are nonvolatile. Membranes of CPE are available in 20 to 40 mil thicknesses in supported and reinforced versions. They are generally unvulcanized and are spliced with solvent adhesives by solvent welding.

Chlorosulfonated Polyethylene

This synthetic rubber is made by the chlorosulfonation of polyethylene. It can be used in both vulcanized and unvulcanized compounds; however, liners of this rubber are generally based on unvulcanized compounds containing at least 45% of the rubber. They are available in sheeting of 30 to 45 mil thicknesses; most are made with fabric reinforcement of either nylon or polyester scrim. Liners of this rubber have good puncture resistance, are easy to seam in the factory or field with solvents, cements, or heat, and have excellent resistance to weathering, aging, oil, and bacteria. Membranes of this material have been used in the lining of pits and ponds where highly acid-contaminated fluids are encountered.

After polyvinyl chloride, this is the most used polymeric material for liners.

Elasticized Polyolefin

Membrane liners of an elasticized polyolefin have been recently introduced. This material is unvulcanized and thermoplastic and can be easily

seamed with heat either in the field or factory. It features excellent resistance to weathering and oils. Films of this material are supplied in 20-foot widths in 20 to 30 mil thickness.

Ethylene-Propylene Rubber (EPDM)

This synthetic rubber is a terpolymer of ethylene, propylene, and a small amount of a diene monomer that introduces double bonds onto the polymer chain. These double bonds are sites for vulcanization of the rubber and, as the unsaturation is in the side chain of the polymer molecule and not in the main chain, ozone, chemical, and aging resistance are excellent. The rubber is compatible with butyl and is often added to butyl to improve resistance of the latter to oxidation, ozone, and weathering. As it is a wholly hydrocarbon rubber like butyl, EPDM has excellent resistance to water absorption and permeation, but has relatively poor resistance to some hydrocarbons. EPDM liners are supplied in vulcanized sheeting of 20 to 125 mils thicknesses, both supported and unsupported. Special attention is required in splicing and seaming this material, as vulcanizable adhesives must be used.

Neoprene or polychloroprene

Neoprene is a synthetic rubber based primarily on chloroprene. It features good weathering and oil resistance and has been used where these properties are required. It is supplied in vulcanized sheeting of 30 to 125 mils thicknesses. As it is a vulcanized rubber, vulcanizing cements and adhesives must be used for seaming.

Polyester Elastomer

This is an experimental thermoplastic rubber which has recently been introduced as a liner material. It has excellent resistance to oils and can be heat sealed. It is supplied in relatively wide sheets of 7 to 10 mils thicknesses.

Polyvinyl Chloride (PVC)

Polymeric membranes based upon PVC are the most widely used flexible liners. They are available in wide sheets of 10 to 30 mils thicknesses; most is used as unsupported film, but fabric reinforcement can be incorporated. PVC compounds contain 30 to 50% of one or more plasticizers to make the films flexible and rubber-like. They also contain 2% of a chemical stabilizer and various amounts of fillers. There is a wide choice of plasticizers that can be used with PVC, depending upon the application and service conditions under which the PVC compound will be used. PVC polymer generally holds up well in burial tests; however, plasticized compounds of PVC films have deteriorated (11,12,13), presumably due to the biodegradability of the plasticizer. Also, some plasticizers are soluble to a limited extent in water (13). On exposure to weather with its wind, sunlight, and heat, PVC liner materials can deteriorate badly due to loss of plasticizer and to polymer degradation. Consequently, they are generally covered. Plasticized PVC films are quite resistant to puncture and relatively easy to splice by solvent welding, adhesives and heat.

## Testing of Polymeric Membranes

Prior to exposure to the wastes, all of the polymeric membranes were subjected to the tests listed in Table 10. Results of these tests are shown in Table 11. The same tests will be performed on liners after exposure to the various wastes.

TABLE 10. TESTING OF POLYMERIC MEMBRANE LINERS

| |
|---|
| Water vapor permeability - ASTM E96. |
| Thickness. |
| Tensile strength and elongation at break, ASTM D412. |
| Hardness, ASTM D2240. |
| Tear strength, ASTM D624, Die C. |
| Water absorption or extraction at RT and 70°C, ASTM D570. |
| Seam strength, in peel and in shear, ASTM D413. |
| Puncture resistance - Fed. Test Method Std. No. 101B, Method 2065. |
| Density, ash, extractables. |

Of particular interest and probable significance for comparison with results obtained after long term exposure to wastes, are the results on water absorption and water vapor permeability which are related properties. Chlorosulfonated polyethylene, neoprene, and chlorinated polyethylene exhibited substantial swelling in water at 70°C, indicating a potential for high swell and permeability on long-term exposure to water and aqueous solutions. On the other hand, elasticized polyolefin and polyester elastomer exhibited low levels of swelling. With respect to water vapor permeability, the materials which exhibited the highest water vapor permeability were PVC and the polyester elastomer. Butyl and elasticized polyolefin were the lowest.

In seam strengths the vulcanized rubbers, i.e., neoprene, EPDM, and butyl gave the lowest values. The PVC seamed with cement adhesives, gave peel adhesion values of approximately 15 pounds per inch. The chlorosulfonated polyethylene and the chlorinated polyethylene, which were also seamed with cements, had peel strengths of 28.5 and 22.5 pounds per inch, respectively. In the peel adhesion test the specimens of the two heat-sealed materials, elasticized polyolefin and polyester elastomer, failed at 21 pounds per inch with breaks in the material, indicating that the true adhesion is higher and that failure in the seams would not be expected in this type material.

## Mounting of Membrane Liner Specimens

All polymeric membrane specimens mounted in the exposure cells have field-type seams incorporated across the width at the center (Figure 4). These seams were either made by the supplier or were made by the contractor

TABLE 11- PROPERTIES OF UNEXPOSED POLYMERIC LINERS

| Item | Test Direction | Test Method | Polymeric Membranes, Matrecon Number | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | 6 | 8 | 11 | 26 | 36 | 43 | 57 | 59 | 75 | 77 | 82 |
| Polymer | | | Chloro-sulfonated poly-ethylene | EPDM | PVC | EPDM | Elasti-cized poly-olefin | Neoprene | Butyl | PVC | Poly-ester Elastomer | CPE | Neoprene |
| Fabric reinforcement | | | Nylon | None | None | None | None | None | Yes | None | None | None | None |
| Thickness, mils | | | 32 | 62 | 30 | 39 | 23 | 30 | 34 | 33 | 7 | 29 | 60 |
| Hardness, instant rdg. | | ASTM D2240 | 79 | 62 | 85 | 59 | 90 | 61 | 74 | 80 | 93 | 87 | 61 |
| 10 sec.rdg. | | | 75 | 58 | 79 | 55 | 87 | 57 | 71 | 71 | 93 | 80 | 57 |
| Tensile strength, psi | With | ASTM D412 | 1770 | 1635 | 2800 | 1935 | 2645 | 1910 | 2145 | 2505 | 6770 | 2055 | 1835 |
| | Across | | 1610 | 1550 | 2550 | 1865 | 2540 | 1660 | 2100 | 2365 | 6765 | 2340 | 1675 |
| Elongation at break, % | With | | 240 | 520 | 350 | 440 | 675 | 330 | 60 | 370 | 560 | 325 | 390 |
| | Across | | 225 | 500 | 350 | 460 | 650 | 310 | 25 | 400 | 590 | 480 | 410 |
| Tensile set, % | With | | 78 | 14 | 90 | 9 | 460 | 8 | 4 | 45 | 340 | 140 | 10 |
| | Across | | 79 | 11 | 103 | 9 | 430 | 6 | 2 | 60 | 370 | 160 | 9 |
| S-100, psi | With | | 990 | 350 | 1355 | 385 | 880 | 490 | -- | 1020 | 2715 | 1240 | 405 |
| | Across | | 895 | 320 | 1205 | 330 | 865 | 430 | -- | 895 | 2455 | 560 | 360 |
| S-200, psi | With | | 1715 | 800 | 1980 | 925 | 975 | 1105 | -- | 1570 | 2880 | 1540 | 875 |
| | Across | | 1445 | 740 | 1740 | 830 | 960 | 970 | -- | 1355 | 2585 | 820 | 705 |
| S-300, psi | With | | -- | 1170 | 2585 | 1440 | 1145 | 1805 | -- | 2160 | 3610 | 1955 | 1410 |
| | Across | | -- | 1110 | 2255 | 1305 | 1150 | 1635 | -- | 1860 | 3315 | 1205 | 1275 |
| Tear strength (Die C), ppi | With | ASTM D624 | 317 | 206 | 374 | 193 | 388 | 171 | 287 | 306 | 911 | 273 | 183 |
| | Across | ASTM D624 | 287 | 211 | 370 | 197 | 369 | 170 | 286 | 287 | 782 | 239 | 178 |
| Puncture resistance, lb. | | Fed.Std.101B(2065) | 34.4 | 51.8 | 37.4 | 32.6 | 26.3 | 30.6 | 26.6 | 37.3 | 29.9 | 43.9 | 53.9 |
| Elong., inch | | Fed.Std.101B(2065) | 0.57 | 1.36 | 0.63 | 1.23 | 0.97 | 1.14 | 0.26 | 0.78 | 1.30 | 0.94 | 1.29 |

continued

TABLE 11 (continued)

| Item | Test Method | Polymeric Membranes, Matrecon Number | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | 6 | 8 | 11 | 26 | 36 | 43 | 57 | 59 | 75 | 77 | 82 |
| Polymer | | Chloro-sulfonated poly-ethylene | EPDM | PVC | EPDM | Elasti-cized poly-olefin | Neoprene | Butyl | PVC | Poly-ester Elastomer | CPE | Neoprene |
| Specific gravity | | 1.343 | 1.173 | 1.275 | 1.169 | 0.938 | 1.477 | 1.286 | 1.280 | 1.236 | 1.362 | 1.480 |
| Ash, % | | | | | | | | | | | | |
| As received | ASTM D297 | 3.34 | 6.75 | 6.19 | 7.63 | 0.90 | 12.21 | 23.40 | 6.94 | 0.38 | 12.56 | 13.19 |
| Dry basis (dried at 105°C) | | 3.35 | 6.78 | 6.20 | 7.67 | 0.90 | 12.30 | 23.46 | 6.97 | 0.38 | 12.56 | 13.21 |
| Water absorption, % | ASTM D570 | | | | | | | | | | | |
| At RT for 7 weeks | | 6.06 | 1.01 | 1.21 | 1.36 | 0.30 | 9.92 | 2.85 | 2.24 | 0.81 | 4.37 | 6.16 |
| 11 weeks | | 6.97 | 1.30 | 1.10 | 1.84 | 0.52 | 13.62 | 3.22 | 2.34 | 1.05 | 5.53 | 8.29 |
| At 70°C for 7 weeks | | 101.19 | 2.86 | 10.36 | 3.67 | 0.56 | 67.63 | 12.97 | 7.01 | 1.00 | 44.21 | 31.78 |
| 11 weeks | | 131.01 | 3.55 | 12.13 | 4.52 | 0.45 | 106.97 | 17.54 | 8.25 | 1.10 | 58.39 | 47.41 |
| Water vapor permeability | ASTM E96-BW | | | | | | | | | | | |
| Thickness, mil | | 28 | 61 | 30 | 38 | 38 | 31 | 33 | 33 | 8 | 30 | 61 |
| Moisture vapor transmittance, g/24 h-m$^2$ | | 0.28 | 0.172 | 1.85 | 0.327 | 0.142 | 0.45 | 0.020 | 4.2 | 10.5 | 0.32 | 0.096 |
| Permeance, metric perms[a] | | 0.024 | 0.015 | 0.16 | 0.028 | 0.012 | 0.039 | 0.0017 | 0.36 | 0.91 | 0.028 | 0.008 |
| Permeance x thickness in cm x 10$^3$ | | 1.7 | 2.3 | 12.2 | 2.7 | 0.85 | 3.1 | 0.15 | 30.2 | 18.2 | 2.1 | 1.2 |
| Seam strength | | | | | | | | | | | | |
| Method of bonding[b] | | Cement | Cement | Solvent | Cement | Heat | Cement | Cement | Solvent | Heat | Solvent | Cement |
| | | | LTV[c] | | LTV[c] | | LTV[c] | LTV[c] | | | | LTV[c] |
| Width of lap seam, in. | | 2 | 2 | 2 | 2 | 0.5 | 2 | 2 | 2 | 0.5 | 2 | 2 |
| Time after seaming, days | | 40 | 39 | 39 | 293 | (d) | 39 | 39 | 39 | (d) | 234 | 39 |
| Peel strength (avg)[e], ppi | ASTM D413 (Modified) | 23.2 | 5.8 | 15.2 | 4.9 | -- | 8.2 | 8.0 | 15.2 | -- | 21.4 | 10.4 |
| Peel strength (max), ppi | | 28.5 | 6.5 | 16.6 | 5.2 | 21.0 | 9.1 | 8.8 | 16.0 | 20.8 | 22.4 | 11.3 |
| Locus of failure[f] | | AD+DEL | AD | AD | AD | SE | AD-LS | AD | AD | SE | AD | AD-LS |
| Shear strength[g] (max),ppi | -- | 62.1 | 29.3 | >62.1 | 39 | 28.8 | 45.7 | >84.8 | >69.1 | >21.2 | 48.3 | 84.9 |
| Locus of failure[f] | | SE | AD | CL | AD | SE | AD-LS | CL | CL | CL | SE | AD-LS |
| % of tensile strength of liner[h] | | 120 | >31 | >81 | 54 | 49 | 92 | >119 | >89 | >45 | 71 | 85 |

[a] Metric perm = 1 g per 24 h per square meter per mm Hg pressure.
[b] See Table 12 for details.
[c] LTV = Low temperature vulcanizing or curing.
[d] Seams made by supplier.
[e] Strip specimen 1 in wide; initial distance between jaws, 2 in; rate of separation of jaws, 2 ipm.
[f] Locus of failure:
CL - Break at clamp
SE - Break at seam edge.
AD - Failure in adhesive.
AD-LS- Failure between adhesive and liner surface.
DEL - Delamination in liner.
[g] Strip specimen 1 in. wide; initial distance between jaws, 4 in; rate of separation of jaws, 2 ipm.
[h] $\frac{\text{Shear value measured at 2 ipm}}{\text{Force to break liner (from tensile strength test at 20 ipm)}} \times 100.$

in accordance with procedures recommended by the respective suppliers. A description of the specific seams incorporated in the liner specimens are given in Table 12.

The polymeric membrane specimens, with the exception of neoprene, were mounted in the cells with a sealing caulk applied on the upper surface of the specimen facing the flange of the tank. A gasket of ¼-inch closed-cell neoprene sponge was placed between the liner specimen and the flange of the base. The sponge gasket compensates for irregularities in the two flanges and allows for a better seal (Figure 1). The neoprene liners were mounted with the same ¼-inch neoprene sponge on the upper side of the liner and no caulk was used.

Two caulking compounds were used in sealing the liners. Since the caulks are exposed to the waste, they were chosen on the basis of our preliminary bench tests. We used a butyl caulk with the acid and caustic wastes and a polysulfide caulk with the other wastes.

After allowing time for the caulk to cure, the cells were tested by filling with water. A couple of the cells leaked slightly, but could be sealed by re-tightening the cell flanges to compensate for the compression set taken by the neoprene foam. One liner had to be completely remounted. One cell that passed the water test and is filled with waste has developed a slight seepage between the liner and the exposure cell tank. It may have to be remounted.

TABLE 12. SEAMS[a] IN EXPOSURE TESTS

| Item | Type of Adhesive | Lap seam width | Fabricator |
|---|---|---|---|
| **Admix Materials** | | | |
| Asphalt concrete | None | -- | -- |
| Emulsified asphalt on nonwoven fabric | Fabric sewn and covered with asphalt. | Sewn | Supplier |
| Fine grain native soil | None | -- | -- |
| Soil cement | None | -- | -- |
| Modified bentonite | None | -- | -- |
| **Polymeric Membranes** | | | |
| Butyl rubber | Vulcanizable adhesive furnished by supplier of liner. | 2 inches | Matrecon |
| Chlorinated polyethylene | Solvent weld with mixture of 1 part toluene and 1 part tetrahydrofuran. | 2 inches | Matrecon |
| Chlorosulfonated polyethylene | Adhesive furnished by liner supplier. | 2 inches | Matrecon |
| Elasticized polyolefin | Heat sealed. | 0.5 inches | Supplier |
| Ethylene propylene rubber | Adhesive and gum tape furnished by supplier | 2 inches | Matrecon |
| Neoprene | Cement and lap sealant furnished by supplier of liner. | 2 inches | Matrecon |
| Polyester | Heat sealed. | 0.5 inches | Supplier |
| Polyvinyl chloride | Solvent weld using mixture of 2 parts tetrahydrofuran and 1 part cyclohexanone. | 2 inches | Matrecon |

[a]All seams were allowed to age at least a month before tested or covered with wastes.

## SECTION 7

## SELECTION AND CHARACTERISTICS OF WASTES

Six classes of hazardous wastes were selected for this investigation. They are:

Acidic sludge
Alkaline sludge
Cyclic hydrocarbon sludge
Lead waste from gasoline tanks
Oil refinery tank bottom waste
Pesticide sludge

Twelve individual wastes were obtained and a preliminary characterization was made of each. Eleven of these wastes met the classifications and are or will be included in the exposure tests.

Limited data on each of the individual wastes are given in Table 13. Both tank bottom wastes were satisfactory for the test; however, neither was received in adequate quantity to place in the required number of cells and a third tank bottom waste was collected. It was not satisfactory and a fourth is now being obtained. It is scheduled for delivery in January 1977.

The wastes were received in 55-gallon drums. Several were found to be inhomogeneous; as they had separated into two or more phases, to insure uniform wastes in the exposure cells, each waste was blended before loading into the individual cells.

TABLE 13. INITIAL CHARACTERIZATION OF WASTES

| Class of Waste | Identification of specific waste | Amount rec'd, drums | pH | Solids, % Total | Solids, % Volatile | Remarks |
|---|---|---|---|---|---|---|
| Strong acid | "HFL" | 1 | 4.8 | 2.48 | 0.29 | -- |
| | "$HNO_3$, HF, HOAC" | 4 | 1.5 | 0.77 | 0.12 | -- |
| Strong base | "Slop water" | 1 | 12.0 | 22.43 | 5.09 | -- |
| | "Spent Caustic" | 4 | 11.3 | 22.07 | 1.61 | -- |
| Tank bottom[a] | "Weed oil" | 1 | 7.5 | 1.81 | 1.00 | Mostly water w/small amount of highly aromatic oil. |
| | "Tergol Clay" | 1 | b | 71.8 | ca 39 | Oil and clay. |
| Lead | "Low lead gas washing" | 1 | 7.2 | 1.52 | 0.53 | 34 ppm lead. |
| | "Gasoline washwater" | 1 | 7.9 | 0.32 | 0.17 | 11 ppm lead. |
| | "Mohawk leaded" | 2 | 7.9 | 0.73 | 0.60 | 5 ppm lead. |
| Saturated and Unsaturated oil | "Oil pond 104" | 5 | b | ca 36 | ca 31 | -- |
| Pesticide | "Weed killer" | 5 | 2.7 | 0.78 | 0.46 | -- |

[a] Four drums of a waste labelled "Aromatic oil waste" were also received, but they were almost entirely water and the "oil" that was present was highly volatile.
[b] Not determined.

## SECTION 8

## EXPOSURE TESTS

### PRELIMINARY EXPOSURE TESTS

It was recognized at the start that some of the wastes could not be contained by some of the liner materials. For example, wastes containing high ion concentrations cannot be contained by bentonite and many of the materials swell badly or dissolve in oily wastes, particularly the asphaltic materials.

Before selecting the combinations of liner materials and wastes to expose, a series of bench tests was run in which small specimens of the liner materials were immersed in the various wastes (Figure 11). For example, 8 different PVC liners were immersed. They all hardened and shrank in slopwater, but not in spent caustic; they shrank in saturated oils and swelled in aromatic oils. Unvulcanized chlorosulfonated polyethylene dissolved in aromatic oils and swelled badly in the other oily wastes. Cured chlorosulfonated polyethylene swelled slightly in oily wastes and two varieties of EPDM swelled in oily wastes. CPE and butyl rubber swelled badly in the oily wastes; neoprene swelled in aromatic oils and hardened in strong acids. The elasticized polyolefin swelled in the oily wastes and polyester liners swelled slightly. A series of polyurethanes were found to swell to varying degrees in the oily wastes. Also included in the preliminary exposure tests were all of the raw materials which might be used in fabricating the exposure cells and in sealing the liners in the cells, i.e the epoxy coating and the various caulks and sealants.

The results of these exposure tests which were run for approximately 3 months are presented in Table 14.

Preliminary exposure tests of the modified bentonite-sand mixtures in contact with the wastes were performed. The test specimens were prepared in accordance with recommendations of the supplier of Clay A. They consisted of a mixture of 20% modified bentonite, 80% well-graded sand placed as a 1-inch deep uncompacted layer in polypropylene beakers having perforated bottoms. They were then covered with water to hydrate the clay. After 5 days the water was removed and the various wastes were poured on (Figure 12). The length of time for the wastes to pass through the uncompacted sand-clay layers was recorded (Table 15).

Strong bases and strong acids percolated through both clays in only a few days. For most of the wastes the percolation times were shorter for Clay A than for Clay B but, in several cases, once percolation had started, it continued at a faster rate in Clay B than in Clay A. On the basis of these tests

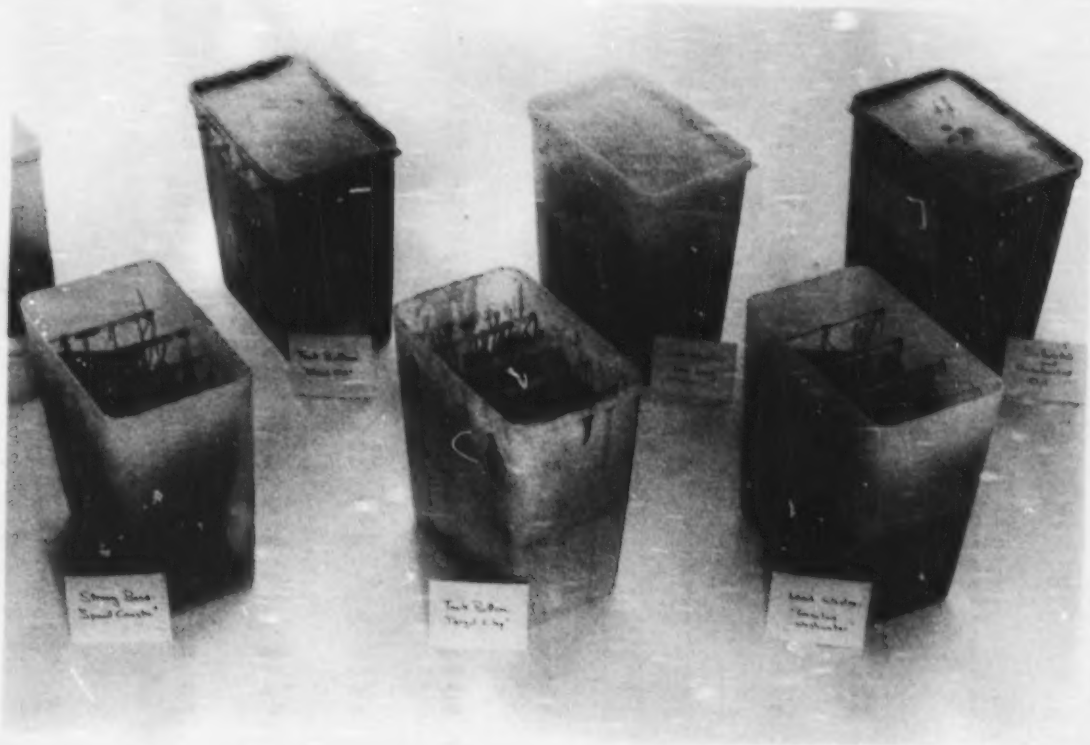


Figure 11. Bench-scale tests of membrane liners partially immersed in hazardous wastes. The polyethylene containers holding weed oil and saturated and unsaturated oil swelled slightly. There was no visible change in the other containers over several months of exposure.

TABLE 14. EFFECT OF IMMERSING LINER AND SEALING MATERIALS IN HAZARDOUS WASTES - A PRELIMINARY STUDY

Unit: % increase in area of portion of strip immersed

| Item | Identification Number | Strong Acid | | Strong Base | | Tank Bottom | | Lead Wastes | | Sat. & Unsat. Oil[e] | Pesticide Weed Killer |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | HFL[a] | $HNO_3$[b] | Slop Water | Spent Caustic | Weed Oil[c] | Tergol Clay | Low Pb Washing[d] | Gasoline Wash Water | | |
| Polyvinyl chloride (PVC) | 10 | CC | NVC | HDS | NVC | NVC | HDS, -17 | NVC | NVC | HDS, -10 | NVC |
| | 11 | NVC | NVC | HDS | NVC | NVC | HDS, -12 | NVC | NVC | HDS, - 5 | NVC |
| | 17 | NVC | NVC | HDS | NVC | +11 | HDS, -10 | NVC | NVC | HDS, - 6 | NVC |
| | 40 | NVC | NVC | HDS | NVC | +24 | SHD | NVC | NVC | SHD | NVC |
| | RM49[f] | -- | -- | -- | -- | +3 | -- | -- | -- | -- | -- |
| | 59 | NVC | NVC | HDS | NVC | +26 | - 6 | NVC | NVC | SHD | NVC |
| | 67 | NVC | NVC | HDS | NVC | +5 | SHD, - 6 | NVC | NVC | SHD | NVC |
| | RM71 | NVC | g | HDS | NVC | CC | HDS, - 8 | NVC | NVC | HDS, - 6 | NVC |
| Chlorosulfonated polyethylene | RM 6[f] | NVC | NVC | NVC | NVC | NVD | NVC | NVC | NVC | 40 | NVC |
| | RC50 | -- | -- | -- | -- | 6 | -- | -- | -- | -- | -- |
| | 55 | NVC | NVC | NVC | NVC | -- | NVC | NVC | NVC | 36 | -- |
| Chlorinated polyethylene (CPE) | 12[f] | NVC | NVC | NVC | NVC | NVD | 6 | NVC | NVC | 11 | NVC |
| | 38 | NVC | NVC | NVC | NVC | NVD 92 | NVC | NVC | NVC | 16 | NVC |
| | RM39 | NVC | NVC | NVC | NVC | NVD[h] | NVC | NVC | NVC | 9 | NVC |
| | RP40 | -- | -- | -- | -- | NVD[h] | -- | -- | -- | -- | NVC |
| | RP73 | NVC | NVC | NVC | NVC | -- | NVC | BLS | NVC | CC, 2 | -- |
| Ethylene propylene rubber (EPDM) | 8[f] | NVC | NVC | NVC | NVC | 75 | 24 | NVC | NVC | 77 | -- |
| | 26 | NVC | NVC | NVC | NVC | 90 | 60 | NVC | NVC | 70 | NVC |
| Polychloroprene (neoprene) | 9 | CC | SCR | CC | NVC | 111 | 77 | NVC | NVC | 42 | -- |
| | 37 | NVC | NVC | NVC | NVC | 67 | NVC | NVC | NVC | 21 | NVC |
| | RM42 | NVC | NVD | NVC | NVC | SEV | NVC | NVC | NVC | 5 | NVC |
| | 43 | -- | -- | -- | -- | 10 | 13 | -- | -- | 48 | NVC |
| | RP47 | -- | -- | -- | -- | 6 | -- | -- | -- | -- | -- |
| | RM56 | NVC | NVC | SHD | NVC | 100 | 3 | NVC | NVC | 10 | NVC |
| | RP74 | NVC | NVC | h | NVC | NVC | NVC | NVC | NVC | NVC | -- |
| Butyl rubber | 22 | NVC | NVC | NVC | NVC | 68 | 64 | NVC | NVC | 69 | NVC |
| | 24 | NVC | NVC | NVC | NVC | 91 | 59 | NVC | NVC | 89 | -- |
| | 44 | -- | -- | -- | -- | 76 | 13 | -- | -- | 94 | -- |
| | RM57[f] | NVC | NVC | NVC | NVC | 67 | 20 | NVC | NVC | 40 | NVC |

(continued)

TABLE 14 (continued)

| Item | Identification Number | Strong Acid | | Strong Base | | Tank Bottom | | Lead Wastes | | Sat. & Unsat. Oil[e] | Pesticide Weed Killer |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | HF[a] | $HNO_3$[b] | Slop Water | Spent Caustic | "Weed Oil"[c] | Terpol Clay | Low Pb Washing[d] | Gasoline Wash Water | | |
| Elasticized polyolefin | 36 | NVC | NVC | NVC | NVC | 49 | 17 | NVC | NVC | 32 | NVC |
| | 41 | NVC | NVC | NVC | NVC | 42 | 18 | NVC | NVC | 24 | NVC |
| Thermoplastic polyester | 69 | NVC | NVC | j | NVC | 12 | NVC | NVC | NVC | NVC | NVC |
| | 75 | NVC | NVC | j | NVC | 18 | NVC | NVC | NVC | 9 | NVC |
| Polyurethane | RP45 | — | — | — | -- | 50 | -- | -- | -- | — | -- |
| | RP46 | -- | — | -- | -- | 44 | -- | -- | -- | -- | -- |
| | RP51 | -- | — | -- | -- | 2 | -- | -- | -- | -- | -- |
| | RM70 | CC | NVC | CC | NVC | 33 | NVC | CC | NVC | 14 | NVC |
| | RP72 | CC | NVC | CC | NVC | 28 | NVC | NVC | NVC | 5 | -- |
| Coal Tar Pitch + PVC | 52 | NVC | NVC | HDS | NVC | 17 | NVC | NVC | NVC | NVC | NVC |
| Emulsified asphalt on nonwoven fabric | 58 | NVC | SCR,BLS | NVC | NVC | NVD | NVD | NVC | NVC | NVD | BLS |
| Butyl caulk | 63 | NVC | NVC | NVC | NVC | NVD | -- | NVC | NVC | — | -- |
| Polyurethane caulk | 64 | -- | NVD | SFT 1&2 | NVC | SFT 1&2 | -- | NVC | BLS | — | -- |
| Polysulfide caulk | 66 | NVC | SFT 1&2 | SCR | NVC | NVC | NVC | NVC | NVC | NVC | -- |
| Teflon sponge rod | 68 | NVC | NVC | NVC | NVC | ABS | ABS | NVC | NVC | ABS | -- |
| Neoprene sponge | 34 | NVC | SCR | SHR | NVC | 38 | SHR | SHR | SHR | SHR | SHR |

[a] Hydrofluoric.
[b] Hydrofluoric, acetic and nitric acids.
[c] "Weed oil" (highly aromatic) 30%; water 70% (tank bottom waste).
[d] Low lead gas washings.
[e] Oil pond 104.
[f] Materials incorporated in cells
[g] 0.10 in. swell in each direction, curled, and delaminated.
[h] Complete loss of tear strength.
[i] Neoprene in satisfactory condition but reinforcing fabric (polyester) dissolved.
[j] Black rubs off, indicating possible dissolving of the liner.

ABBREVIATIONS:
ABS - Absorbed waste.
BLS - Blistering of surface.
CC - Color change.
DEL - Delaminated

ABBREVIATIONS, contd.
DIS - Dissolved or disintegrated.
HDS - Hardened and shrank.
NVC - No visible change.
RC - Reinforced and cured.
REV - Reverted.
RF - Reinforced with fabric.
RN - Reinforced with nylon.
RP - Reinforced with polyester.
NVD - Removed specimen from waste because of excessive swelling or disintegration.
SCR - Surface cracking and hardening.
SFT-1 - Softened above waste.
SFT-2 - Softened in waste.
SHD - Slightly hardened.
SHR - Shrank.
SS - Slightly swollen.

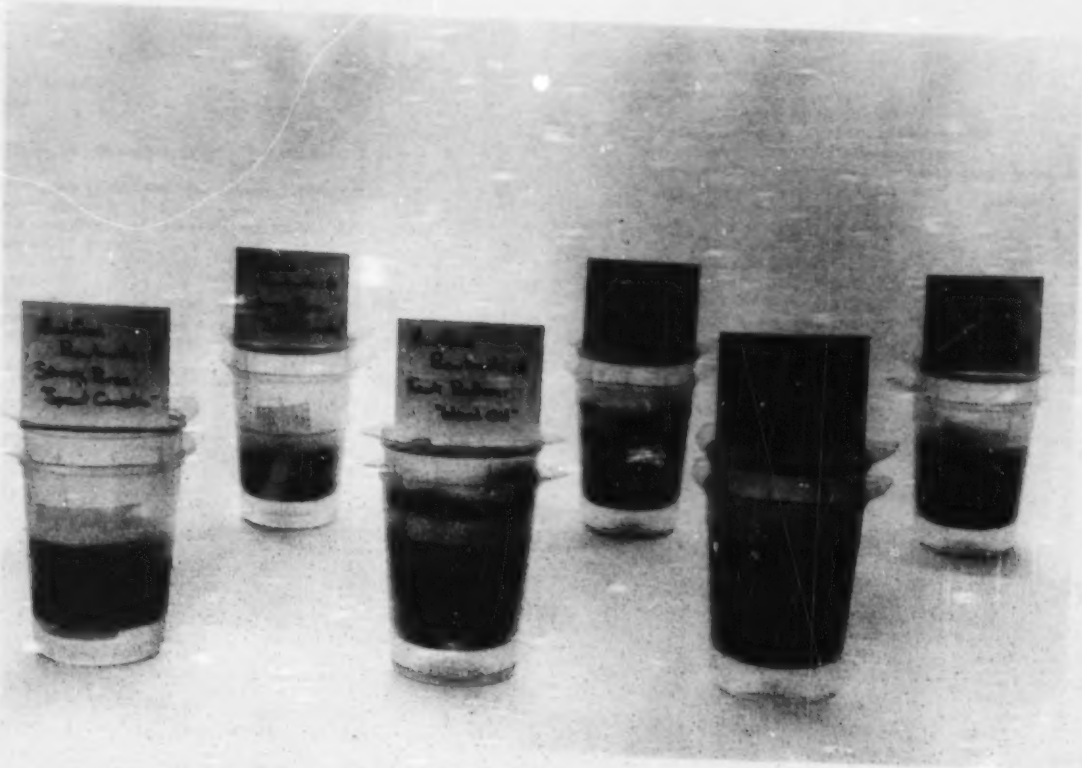


Figure 12. Bench-scale test of modified bentonite clays exposed to various hazardous wastes. The inner beaker has been perforated and filled with a sand bentonite clay mixture. The mixture was covered with water for 5 days before the waste was added.

TABLE 15. EFFECTS OF PLACING HAZARDOUS WASTES ABOVE TEST SPECIMENS OF ADMIX MATERIALS

Time to first passage of wastes through material, in days

| Item | Strong Acid | | Strong Base | | Tank Bottom | | Lead Wastes | | Sat. & Unsat. Oil[e] |
|---|---|---|---|---|---|---|---|---|---|
| | HNO$_3$[a] | HNO$_3$[b] | Slop Water | Spent Caustic | Used Oil[c] | Terpol Clay | Low Pb Washing[d] | Gasoline Wash Water | |
| Treated bentonite-sand mixtures | | | | | | | | | |
| A | 17 | 3 | 2 | 3 | 32 | > 47 | > 47 | > 47 | 14 |
| B | > 44 | 3 | 2 | 3 | > 47 | > 47 | > 47 | > 47 | > 47 |
| Native soil (Mare Island) | — | > 19 | > 19 | 11[f] | — | — | — | — | — |
| Soil-cement, containing rice hull ash | — | > 19[g] | > 19[j] | 2[h] | — | — | — | — | — |
| Soil-cement, portland | > 19 | > 19[i] | > 19[j] | [j] | — | — | — | — | — |

[a-e] See notes on Table 14.
[f] Some dampening of bottom; neutral to litmus.
[g] Bubbling on surface.
[h] Fluid passing through is basic.
[i] Surface softer.
[j] NVC - no visible change to surface of specimen in 19 days.

we dropped the combinations of modified bentonite with the acidic and caustic wastes for exposure tests in the cells.

Soil and soil cement which had been selected for the study were exposed to the various wastes by sealing 2-inch diameter cylindrical specimens in one end of 8-inch long, 54 mm i.d. Pyrex cylinders using Epoxy 1 and Epoxy 4. 3 days after the epoxy cement was cast the cylinders were filled with the wastes to a depth of 5 inches.

The fine-grain soil specimens (Figure 13) were prepared by molding the soil at a water content of 20 parts per 100 parts dry soil in a 2-inch diameter mold. The soil was compacted in 5 lifts using a high-frequency impact tool followed by double-end static compaction. The height of the specimens was about 2 inches.

The specimen in acidic waste, "16% $HNO_3$", released a few bubbles immediately, and continued to release bubbles slowly for about a week. Within 1 day the surface of the specimen developed a flaky appearance and the epoxy peeled off the glass in both "16 % $HNO_3$" and "spent caustic"; within 2 days the same effect was noted with the specimen in "slopwater".

When probed with a glass rod after 7 days of exposure, the specimen exposed to "slopwater" had a hard surface; the specimen exposed to "spent caustic" was soft on the surface, and the surface of the specimen exposed to "16% $HNO_3$" became flaky. There was no leakage from any of the specimens. After 11 days of exposure a slightly damp spot appeared at the center of the bottom of the specimen exposed to "spent caustic". After 16 days the damp spot remained unchanged and was neutral to litmus.

The soil cement specimens were molded in a 2-inch diameter mold, with compaction in 5 lifts using a high-frequency impact tool, followed by double-end static compaction, of the following compositions:

| | | |
|---|---|---|
| "Wastes fines" from rock crushing | 100 | 100 |
| Portland cement | 10 | -- |
| Rice hull ash cement | -- | 10 |
| Water | 12 | 12 |

Three specimens of each, approximately 2 inches high, were fastened into 1 end of Pyrex cylinders. All specimens had been kept moist for 3 weeks to cure. 3 days after the epoxy cement was cast, the cylinders were filled to a depth of 5 inches with "spent caustic", "slopwater", and "16% $HNO_3$", respectively. "16% $HNO_3$" released a few bubbles from both specimens.

After 1 day, "16% $HNO_3$" had caused the epoxy to peel off the glass in the thin layer exposed above the specimens, and a surface layer of soil cement had flaked off slightly on the rice hull ash cement specimen and a little more on the portland cement specimen. A few bubbles continued to form. The specimens exposed to "spent caustic" and "slopwater" appeared unchanged. Concrete based on the rice hull ash cement has been reported to have acid resistance.

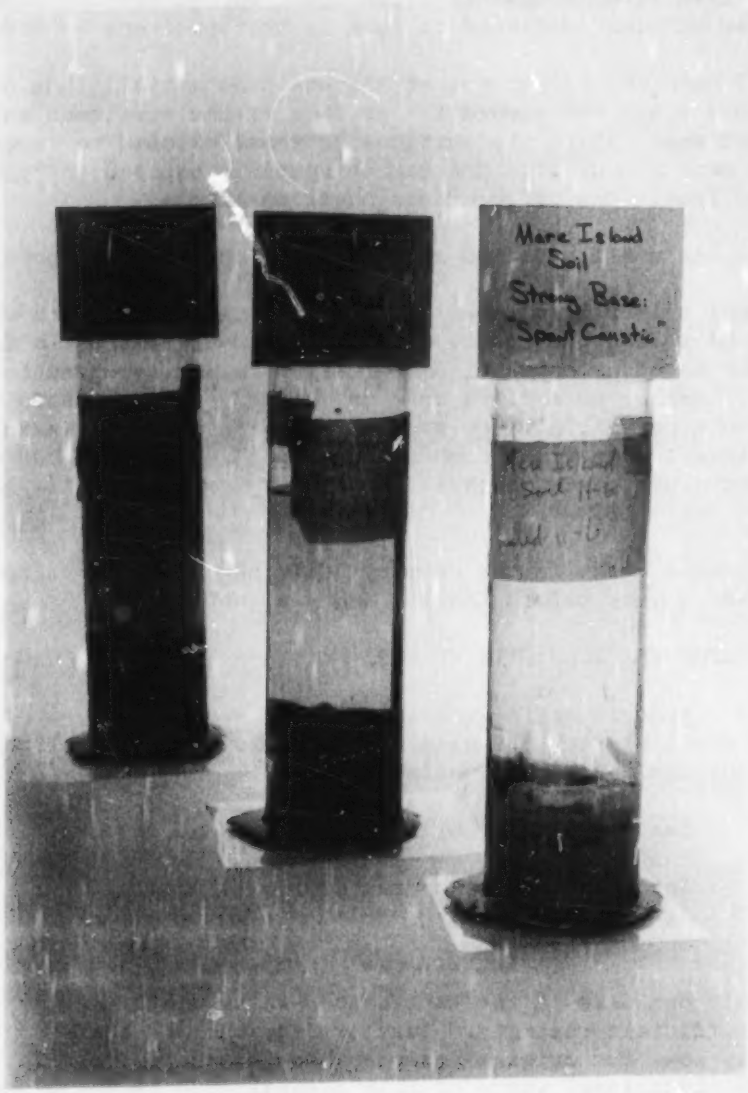



Figure 13. Bench-scale test of compacted soil samples exposed to hazardous wastes. The compacted cylindrical sample was sealed in the bottom of an open-ended glass cylinder with an epoxy resin and 5 inches of waste placed on top. Soil cement specimens were tested in the same manner.

After 2 days some liquid began to seep through the RHA cement specimen exposed to "spent caustic". The thin layer of epoxy peeled off the glass in the cylinders filled with "slopwater", but there was no visible change in the specimens. A few bubbles continued to form in the specimens exposed to "16% $HNO_3$".

After 1 week the appearance of all was substantially the same as above. Probing with a glass rod showed the surface of the specimens exposed to "16% $HNO_3$" had softened, while the surfaces of those exposed to "spent caustic" and "slopwater" were sound. The RHA cement specimen exposed to "spent caustic" continued to leak. The effluent was basic to litmus.

After 2 weeks all appeared substantially the same as above.

The final selection of combinations of liners and wastes was made so that there would be reasonable chance of a long exposure without a failure. For example, liners based on asphaltic materials were not selected for confining oily wastes; neither were clays used to confine strong caustic or acidic wastes. When a polymeric liner swelled badly in aromatic waste, that combination was deleted. The final combinations of liners and wastes which were selected for exposure tests, both in the primary exposure cells and in open tubs, are shown in Table 16.

The exposure tests in the primary cells have been initiated and will require at least a year before results are available.

FILLING OF EXPOSURE CELLS WITH WASTES

When the exposure cells were loaded with wastes, only 5 of the 6 types of wastes were available in sufficient quantities, i.e. 200 gallons or 4 drums, to fill the desired number of cells:

Lead wastes (by blending 3 different wastes)
Pesticide
Saturated and unsaturated oils
Strong acid
Strong base

There was only one drum of the tank bottom waste containing aromatic oils and this was insufficient quantity. Furthermore, the oil content of this waste was extremely low; the original samples had apparently been taken from the top of the drum. Additional waste of this general type is being obtained.

All of the wastes were received in closed 55-gallon drums and were much lower in viscosity than had been anticipated. It was originally expected that they would be sludge-like and could be handled with buckets from open-top drums. Furthermore, some were found to be very inhomogeneous, consisting of 2 liquid phases and particulate solids. Since the waste of a given type should have the same composition in all the cells in which it is placed, it was decided that each waste should be thoroughly mixed and blended before being added to the cells. It was also decided to pump the wastes from drum to

TABLE 16. COMBINATIONS OF LINER MATERIALS AND WASTES SELECTED FOR EXPOSURE TESTING IN CELLS AND OPEN TUBS - NUMBER OF CELLS MOUNTED WITH LINERS.

| Item | Strong acid | Strong base | Lead waste | Sat. and unsat. oil | Pesticide | Tank Bottom Aromatic oil | Total No. of Cells w/liners |
|---|---|---|---|---|---|---|---|
| Admix Liners | | | | | | | |
| Asphalt concrete | 2 | 2 | 2 | - | 2 | - | 8 |
| Emulsified asphalt on nonwoven fabric | 2 | 2 | 2 | - | 2 | - | 8 |
| Fine grain native soil | - | 2 | 2 | 2 | 2 | 2 | 10 |
| Soil-cement | - | 2 | 2 | 2 | 2 | 2 | 10 |
| Modified bentonite | - | - | 2 | 2 | 2 | 2 | 8 |
| Polymeric Membranes | | | | | | | |
| Butyl rubber | 2 | 2[a,b] | 2 | - | 2 | - | 8 |
| Chlorinated polyethylene | 2 | 2a | 2 | 2 | 2 | - | 10 |
| Chlorosulfonated polyethylene | 2[a] | 2[a,b] | 2 | 2 | 2 | - | 10 |
| Elasticized polyolefin | 2 | 2[a] | 2 | 2[a] | 2 | 2 | 12 |
| Ethylene propylene rubber | 2[a] | 2[a] | 2 | - | 2 | - | 8 |
| Neoprene | - | 2 | 2 | 2[a] | 2 | - | 8 |
| Polyester | 2 | 2 | 2 | 2[a] | 2 | 2 | 12 |
| Polyvinyl chloride | 2[a] | 2 | 2 | 2[a] | 2 | 2 | 12 |
| Total cells per waste | 18 | 24 | 26 | 18 | 26 | 12 | 124 |

[a]Combinations exposed in open tubs.
[b]"Slopwater".

drum and into the individual exposure cells, thus reducing the chances for spillage. The equipment which was set up to mix and pump these wastes is shown in Figure 14.

It was necessary to obtain a pump which was compatible with the wastes, self-priming, able to pump solids in suspension, and had sufficient capacity to transfer the contents of a 55-gallon drum in 10 minutes. A flexible impeller pump was obtained having an epoxy pump body and a totally enclosed motor which allowed hosing down of the pump without getting water into the motor. The rated capacity was 7 gallons per minute. Both the impeller and the pump body were replaceable in the event either was damaged by the wastes.

The plan for the mixing and blending of each waste was:

1. To mix thoroughly the contents of each drum by pumping them into an open-top drum equipped with a 1750 rpm mixer with dual propellers and mixing with the remaining solids flushed from the original 55-gallon drum.

2. To transfer ¼ of the contents to each of 4 additional open-top drums for blending.

3. To accumulate the mixed quantities in the open-top blending drums and mix each drum thoroughly.

4. To pump the wastes from the open-top blending drums into the individual cells.

Thus, each exposure cell could receive an aliquot of the waste of the same composition as that received by every other cell containing that waste.

The wastes were loaded in the cells in the order of anticipated increasing difficulty, based on viscosity and the apparent amount of solids. The "pesticide" having the consistency of water was processed first without problems using the flexible impeller pump.

The "spent caustic" appeared to be the next easiest waste to handle, as it was less corrosive than the strong acid ("$HNO_3$") waste and appeared to be an aqueous solution. However, before 2 drums of caustic had been mixed and transferred, undissolved salts wore away the epoxy body and face-plate of the pump which ceased to operate. To solve this problem a second pump, a centrifugal type of 20 gallons per minute, was obtained. The impeller and housing are made of cast iron which is more resistant to the abrasion of the salts in the caustic waste than was the epoxy body of the first pump. Furthermore, the centrifugal pumping action does not depend upon close fitting parts and will handle particles up to 3/8 inch in diameter.

A strainer was placed over the end of the inlet pipe to strain large particles when transferring from the closed-head drum. These large particles were eventually broken-up manually to a pumpable size, and flushed out of the drum. The shearing action of the mixer in the open-head drum further disintegrated these insoluble salts.

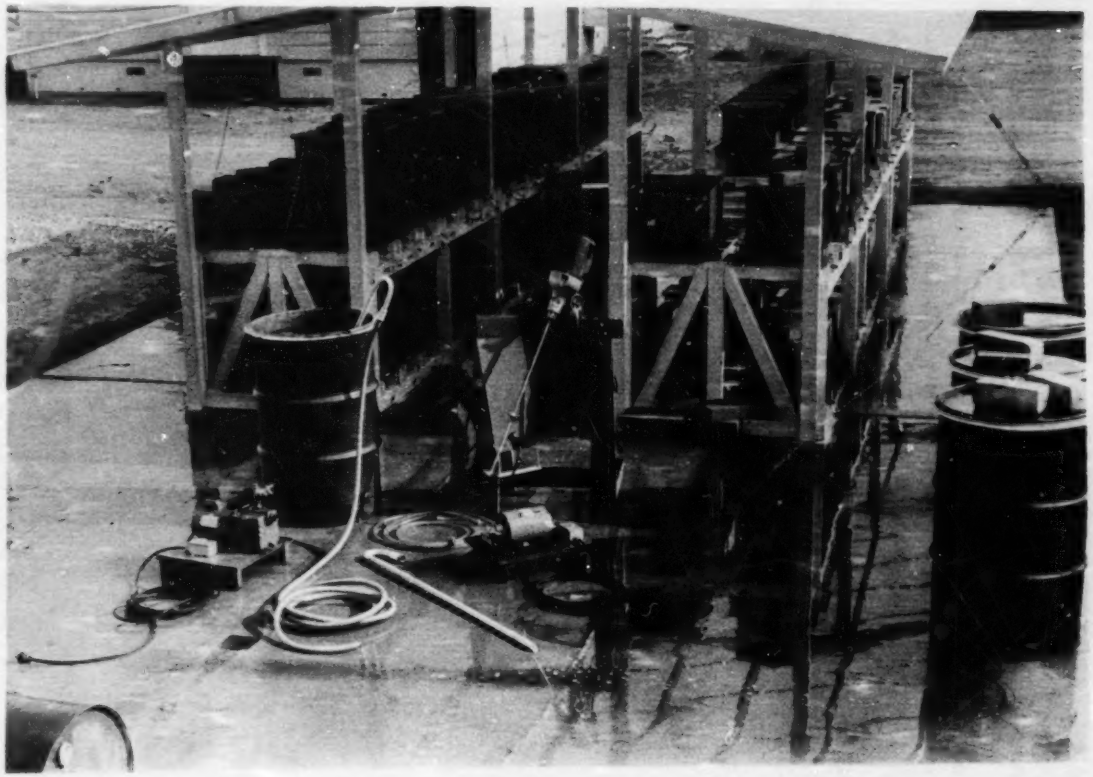


Figure 14. Equipment used in the blending of wastes and filling of cells.

To fill the 5-gallon individual exposure cells using the 20 gpm contrifugal pump, it was necessary to reduce the flow by putting a "T" in the system and circulating a portion of the waste. This pumping system worked very well with the caustic waste.

There was an insufficient amount of a single lead waste for filling the primary exposure cells; consequently, the 3 different lead wastes which were available were combined by the mixing and blending process described above to yield the 4 drums needed to fill the exposure cells. The centrifugal pump was used in the mixing, blending, and transferring of the lead wastes.

The saturated and unsaturated oil waste was viscous and had to be pumped with the flexible impeller pump. Pumping and blending this oily waste was speeded slightly by warming it with a mantle drum heater.

As the flexible impeller pump is resistant to acids, it was used to pump the "$HNO_3$" waste. The acid would have attacked the cast iron of the centrifugal pump, changing both the character of the waste and of the pump.

## SECTION 9

## OUTDOOR WEATHERING TESTS OF MEMBRANE LINERS

The primary objective in this study is to determine the effects of exposure of various liner materials to hazardous wastes. The major effort of the project is to expose the various liners to the wastes in cells under 1 foot of various wastes to simulate a liner at the bottom of a pit or a pond.

In many actual installations, however, liners are simultaneously exposed to weather, such as on the berm around a pit, as well as to fluid being impounded. Under such situations liners are exposed to ultraviolet and heat of the sun, to wind, to ozone, to variations in temperature, and to rain. These conditions can cause many organic and polymeric materials to degrade, resulting in softening or hardening and possibly in breaking.

Liners which are sensitive to these conditions are covered to avoid the adverse effects of weather. In this project it is desirable to know the full characteristics of liners, including some of their basic limitations. Consequently, weather exposure tests have been included in the program though this type of testing is limited.

Two types of outdoor exposure have been undertaken, both of which involve exposing specimens on the laboratory roof. In the first type, small specimens of polymeric membranes are being exposed on a rack placed at a 45° angle to the south. Three 6 inch by 6 inch specimens of each of 11 polymeric membranes are being exposed (Figure 15). Included are the following materials:

Butyl rubber, fabric reinforced
Chlorinated polyethylene
Chlorosulfonated polyethylene, fabric reinforced
Elasticized polyolefin
Ethylene propylene rubber (2 liners)
Neoprene (2 liners)
Polyester elastomer
Polyvinyl chloride (2 liners)

During the exposure we expect to determine:

- Weight loss incurred
- Dimensional changes
- Surface changes including cracking and checking
- Changes in properties such as hardness, tensile, puncture resistance, and brittleness.

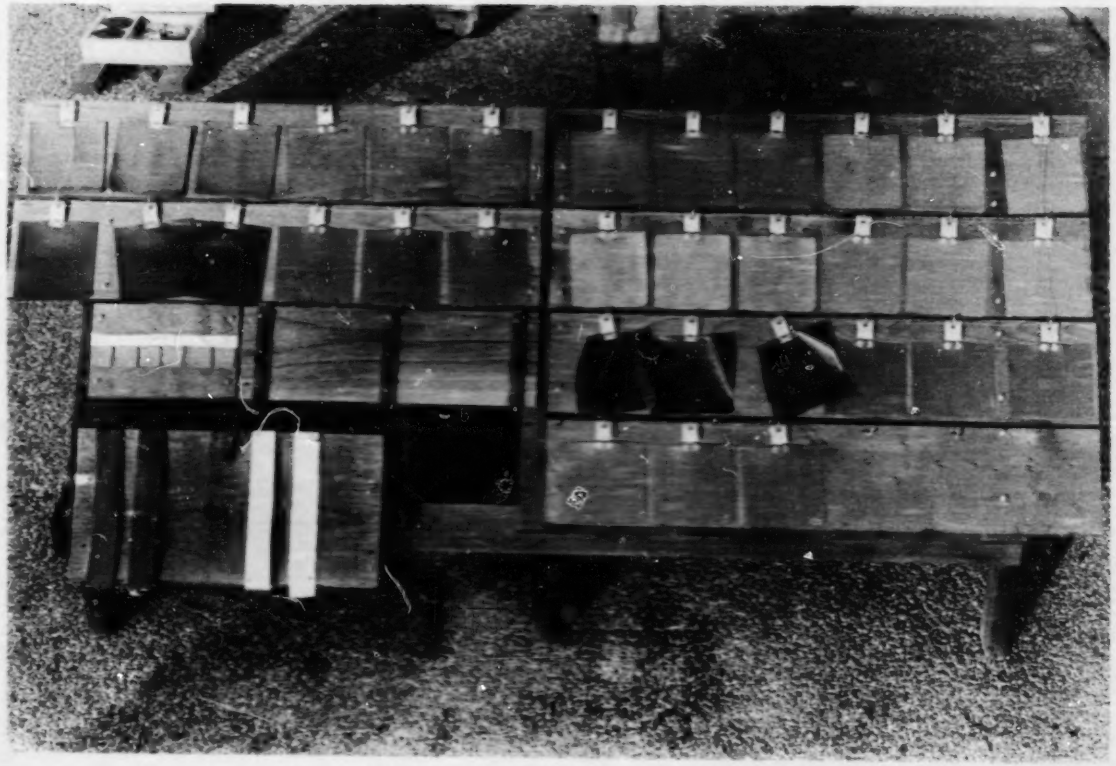


Figure 15. Exposure rack loaded with polymeric membrane specimens. The rack is exposed at a 45° angle to the south.

In the second type of outdoor exposure test small open tubs have been lined with the various polymeric membranes and partially filled with hazardous wastes. These liner specimens are 4 feet by 4 feet and have been folded over the edges and corners of the tubs. Each tub has been covered with chicken wire (Figure 16) to prevent bird bathing. A seam has been incorporated in each of the liners except the polyester elastomer. (The supply of liners with seams which had been received from the manufacturer was exhausted. The material requires special heat sealing conditions which were not available.)

The combinations of liners and wastes which were selected for this test are shown in Table 16. The cells are approximately half filled with wastes; the levels of the wastes in the tubs are allowed to change within limits due to evaporation and rain. The cells can be covered if there is too much rain; if there is excessive evaporation, water is added.

These tubs will be left exposed during the remainder of the project, except in case of failure of a liner. During exposure the overall effects on the liners will be observed, such as swell and cracking, particularly at folds and corners; also, it will be observed if there is any opening of the seams. At the end of the exposure period, when the tubs are emptied and the liners retrieved, the physical properties of the membranes and the seams at various positions will be determined.

Figure 16. The open exposure tubs lined with polymeric membranes and partially filled with hazardous wastes. They are covered with chicken wire and placed in a shallow basin lined with an elasticized polyolefin membrane. During rainy weather these cells are protected by a corrugated plastic cover.

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## APPENDIX A

### MATERIALS USED IN CONSTRUCTING EXPOSURE CELLS AND MOUNTING LINER SPECIMENS

| Material | Use | Trade Name | Supplier |
|---|---|---|---|
| Epoxy Resins: | | | |
| Epoxy 1 | To cast epoxy rings[a] to seal liners cells. | Concresive 1217 | Adhesive Engineering |
| Epoxy 2 | To coat inner walls of cells and bases and to cast sealing rings. | Concresive 1305 | Adhesive Engineering |
| Epoxy 3 | To cast epoxy rings[a] | Concresive 1310 | Adhesive Engineering |
| Epoxy 4 | To seal 2 in. cylindrical specimen in glass cylinder. | Concresive 1001 | Adhesive Engineering |
| Paints: | | | |
| Primer | Coating of inner surfaces of cells. | Koropon CA | De Soto, Berkeley,CA |
| Caulks: | | | |
| Butyl rubber | Seal membrane specimens in cells. | Sure-Seal | Carlisle |
| Polysulfide | Seal membrane specimens in cells. | Colma Joint Sealer (Sikaflex 412) | Sika, Lyndhurst, N.J. |
| Gasket: | | | |
| Neoprene sponge | To seal liners in primary exposure cells. | -- | Cal Neva, Oakland, CA |
| Silica gravel | Inert gravel in bases of cells . | -- | G.E. Dodson Auburn, CA |
| Sandblasting sand | Used in epoxy cast rings and on inside surfaces of spacers. | Clementina No.2 | Clementina Berkeley,CA |

[a]The first rings were made with Epoxy 1; however, as Epoxy 2 and 3 are more resistant to chemicals and solvents, we changed to them.

## APPENDIX B

## MAJOR EQUIPMENT

Paint sprayers:

Binks Model 69 - general purpose standard production sprayer.

Binks 80-210 Quart pressure cup with air regulator.

Binks 66 by 66 SD nozzle with 565 needle (for primer).

Binks 68 by 68 PB nozzle (for epoxy coating).

Air compressor (rented).

Mixers:

½ HP, Model D14 - 1750 rpm, MixMor, Inc., with two 3-inch propellers.

Pumps:

Centrifugal Pump - Teel, Model 3P577A, Dayton Electric

Flexible Impeller Self-Priming Pump, Flotec Model F4P1-1104 - Flotec, Inc. (Neoprene impeller and epoxy body), 1/3 HP, 115V, 1725 rpm.

Soil Shredder:

Model 2-E, W-W Grinder Corp.

## APPENDIX C

## MATERIALS FOR ADMIX LINERS

| Material | Supplier |
|---|---|
| **Soils:** | |
| Fine-grain, high silica content | Mare Island Naval Shipyard, Vallejo, CA. |
| **Soil cement:** | |
| Soil: "Wastes fines" from rock crushing | Quarry Products Company, Richmond, CA. |
| Cement: Type 1-2-5 Portland cement | Kaiser Permanente |
| Rice hull ash (RHA) cement | Industrial Materials Co., Los Angeles, CA. |
| Coating C: Coal tar epoxy, Ceilcote Flake Prime and Flake Tar | Ceilcote |
| Coating K: Coal tar epoxy, Bitumastic 300M | Koppers |
| **Hydraulic asphalt concrete (HAC):** | |
| Hot mix | Ransome Company, Emeryville, CA. |
| Aggregate, dense-graded ½ inch maximum | Lone Star Quarry, Livermore, CA. |
| Asphalt, AR-4000 | Douglas Oil Company |
| **Modified bentonite:** | |
| Sand, dense-grade No. 2 (contained 1% water) | Topsoil King, Richmond, CA. |